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Bionetics Corp., Cincinnati, OH

Prepared for

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16. ABSTRACT

An interlaboratory collaborative study was conducted to determine the precision and accuracy of Method 3050 for the analysis of 23 elements in sediments, sludges and soils. Method 3050 is entitled, "Acid Digestion of Sediments, Sludges and Soils". It includes instructions for quality control, sample preparation and analysis of samples by AA direct aspiration.

The study design was based upon Youden's non-replicate plan for collaborative tests of analytical methods. Initially, 14 solid wastes were collected, direid, homogenized and analyzed for the 23 elements of interest by Inductively-Coupled Plasma. Based upon this data, seven solid wastes were selected that contained the most naturally-occurring elements over a relatively wide concentration range. Elements not occurring naturally were spiked into each solid waste, and subsequently digested with nitric acid and hydrogen peroxide, refluxed with nitric or hydrochloric acid, and analyzed by atomic absorption - direct aspiration. The resulting data were analyzed using USEPA computer program routine entitled, "Interlaboratory Method Validation Study". The data analysis produced measures of precision and mean recovery for the mean recovery for the acid digestion/AA direct aspiration for each element.

The study was conducted under the auspices of the U.S. Environmental Protection Agency, Quality Assurance Research Division, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

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USEPA METHOD STUDY 37
SW-846 METHOD 3050
ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

by

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents and wastes. The Environmental Monitoring Systems Laboratory (EMSL)-Cincinnati, conducts research to:

> Develop and evaluate methods to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
>
> Investigate methods for the concentration, recovery, and identification of viruses, bacteria, and other microorganisms in water.
>
> Determine the response of aquatic organisms to water quality.
>
> Support an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastes.

This publication of EMSL-Cincinnati, entitled EPA Method Study 37, SW-846 Method 3050, Acid Digestion of Sediments, Sludges and Soils, reports the results of EPA's interlaboratory method study for the analyses of 23 trace elements in solid wastes.

Federal agencies, states, municipalities, universities, private laboratories, and industry should find this evaluative study of assistance in monitoring and controlling pollution in the environment.

Thomas A. Clark, Director
EMSL - Cincinnati
November 1988

ABSTRACT

An interlaboratory collaborative study was conducted on SW-846 Method 3050, "Acid Digestion of Sediments, Sludges, and Soils", to determine the overall and single-analyst precision for the analyses of 23 elements in sediments, sludges, and soils. SW-846 Method 3050 includes instructions for quality control, sample preparation and analysis of samples by Flame Atomic Absorption (FLAA) and Graphite Furnace Atomic Absorption (GFAA).

The study design was based upon Youden's non-replicate plan for collaborative tests of analytical methods (1). Initially, fourteen solid wastes were collected, dried, homogenized and analyzed by Inductively Coupled Plasma (ICP) for the 23 metals of interest. Based upon these data, seven solid wastes were selected that contained naturally occurring elements over a relatively wide concentration range. Elements not occurring naturally were added (spiked) into each solid waste. In the formal study by eight laboratories, the solid wastes were digested with nitric acid and hydrogen peroxide, refluxed with nitric or hydrochloric acid, and analyzed for 21 elements by FLAA and for two elements, arsenic and selenium, by GFAA. The results were analyzed using USEPA computer programs entitled "Interlaboratory Method Validation Study" (IMVS) (2). The computer programs produced measures of overall and single-analyst precision for the 21 elements analyzed by FLAA and for the two elements analyzed by GFAA.

The study was conducted by The Bionetics Corporation under the direction of the Quality Assurance Research Division, Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Cincinnati), under Contract No. 68-03-3254. Analytical work was completed as of August, 1987. The study report covers a period from January 10, 1987 to February 5, 1988.

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SECTION 1

INTRODUCTION

The Hazardous Waste Management facility permit regulations promulgated in July, 1982 (40 CFR 265) provide performance standards for the monitoring of ground waters, wastewaters and solid matrices at hazardous waste sites. To facilitate these standards, chemical and physical analyses are required to assess the degree of contamination at and around the area of the site. The Manual: Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, (SW-846), November 1986, Third Edition, provides a unified, up-to-date source of information on sampling and analyses related to compliance with Resource Conservation and Recovery Act (RCRA) regulations. The success of these pollution control activities, particularly when legal action is involved, depends upon the reliability of the data generated by the laboratories, therefore it is important to evaluate the methods through interlaboratory method validation studies.

The Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Cincinnati), develops/selects analytical methods and provides quality assurance (QA) support to agency programs involving water and waste regulations. In EMSL-Cincinnati, the responsibility for providing QA support is assigned to the Quality Assurance Research Division (QARD). Its QA program is designed to establish the reliability and legal defensibility of water and waste data collected by the Agency, the state regulating authorities, the private sector, and commercial laboratories performing compliance analyses. One of its QA activities is to conduct interlaboratory method validation studies to evaluate analytical methods selected for the Agency's operating program such as the Office of Solid Waste.

This report describes an interlaboratory method validation study for SW-846 Method 3050, "Acid Digestion of Sediments, Sludges, and Soils". The elements: aluminum, antimony, barium, beryllium, calcium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, silver, sodium, thallium, vanadium and zinc were analyzed by flame atomic absorption spectroscopy (FLAA) while arsenic and selenium were analyzed by graphite furnace atomic absorption spectroscopy (GFAA). Originally, silver and antimony were included in Method 3050 but were removed from the September 1986 revision of the method. They were, however, included in this study.

The primary objective of the study was to characterize the behavior of Method 3050 in terms of overall precision and single-analyst precision. The study was conducted with the cooperation of eight participating laboratories under the direction of the Quality Assurance Research Division, EMSL-Cincinnati. As primary contractor to QARD, The Bionetics Corporation was responsible for the collection and characterization of the solid matrices, preparation of user instructions and report forms, distribution of samples, and screening the returned data for gross errors. The raw data were evaluated statistically by the QARD using a series of computer programs entitled, "Interlaboratory Method Validation Studies" (IMVS). Upon review of the draft report by EMSL-Cincinnati, The Bionetics Corporation prepared the final report.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

SW-846 Method 3050 is recommended for the analyses of aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, sodium, thallium, vanadium, and zinc by flame atomic absorption and for the analyses of arsenic and selenium by graphite furnace atomic absorption. The linear regression equations obtained from this study, and presented in Table 1, can be used to predict the overall and single-analyst precision of these elements at concentrations in the ranges investigated in this study.

Originally, silver and antimony were included in Method 3050 but were removed from the September 1986 revision of the method. They were, however, included in the study but are not recommended for analyses by Method 3050.

Barium was the only element for which data variability were so great among solid wastes that linear relationships could not be established. As a result, linear regression equations are not reported. Precipitation reactions were hypothesized as a possible cause, but were not confirmed.

The overall precision of Method 3050 for the FLAA elements, expressed as %RSD, ranged from 5.6% for lead to 22.4% for aluminum and potassium and averaged 9.4%. The single-analyst precision of Method 3050 for the FLAA elements, expressed as %RSD-SR, ranged from 2.8% for thallium to 8.6% for aluminum and averaged 5.4%. The ratio of the average overall precision/single-analyst precision was 1.7:1.

The overall precision values as %RSD for the GFAA elements, arsenic and

TABLE 1. REGRESSION EQUATIONS FOR OVERALL (S) AND SINGLE-ANALYST (SR) PRECISION FOR SW-846 METHOD 3050

Element (Conc. Range)	Regression Equation (a)	Waste No. Deleted (b)	Data Set Corrected for Waste No. (c)
Aluminum	S = 0.210X + 51.0		4A,4B
(715-18700 ug/g)	SR = 0.0742X + 41.2		
Antimony	S = 0.145X + 10.58	1,7	
(63-1700 ug/g)	SR = 0.0310X + 16.35	1,7	
Beryllium	S = 0.0676X + 0.37		
(5.5-231 ug/g)	SR = 0.0411X + 0.26		
Cadmium	S = 0.0437X + 0.78	6,7	1A,1B,2B
5.6-154 ug/g)	SR = 0.0446X + 0.41	6,7	
Calcium	S = 0.0893X - 66	7	5B
(11900-185000 ug/g)	SR = 0.0662X - 881	7	
Chromium	S = 0.0923X + 0.73	6	
(67-97700 ug/g)	SR = 0.0564X + 0.66	6	
Cobalt	S = 0.0525X + 7.22	6	
(91-624 ug/g)	SR = 0.0428X - 0.40	6	
Copper	S = 0.0636X + 0.0		7B
(66-25800 ug/g)	SR = 0.0460X + 2.0		
Iron	S = 0.0522X + 693		
(16900-123400 ug/g)	SR = 0.0364X + 162		
Lead	S = 0.0607X - 17.4	2,5	
(715-20800 ug/g)	SR = 0.0292X + 4.3	2,5	
Magnesium	S = 0.0572X + 106		
(2050-50700 ug/g)	SR = 0.0408X + 26		
Manganese	S = 0.0840X - 3.0		
(287-1100 ug/g)	SR = 0.0618X - 6.2		
Molybdenum	S = 0.0739X - 3.5	1	All data sets corrected except 5A,5B
(180-5600 ug/g)	SR = 0.0936X - 10.8	1	

(Continued)

TABLE 1. REGRESSION EQUATIONS FOR OVERALL (S) AND SINGLE-ANALYST (SR) PRECISION FOR SW-846 METHOD 3050 (Continued)

Element (Conc. Range)	Regression Equation [(a)]	Waste No. Deleted [(b)]	Data Set Corrected for Waste No. [(c)]
Nickel (22-75000 ug/g)	$S = 0.0678X + 6.44$ $SR = 0.0437X + 5.64$		2A,2B,7B
Potassium (448-4100 ug/g)	$S = 0.196X + 61.8$ $SR = 0.0739X + 16.1$		
Sodium (227-105700 ug/g)	$S = 0.0646X + 26.4$ $SR = 0.0481X + 8.4$		
Thallium (129-2400 ug/g)	$S = 0.0559X + 7.5$ $SR = 0.0213X + 4.3$		
Vanadium (334-7420 ug/g)	$S = 0.133X - 4.2$ $SR = 0.0437X + 13.6$		6B
Zinc (93-188900 ug/g)	$S = 0.0832X + 1.10$ $SR = 0.0639X + 1.33$	7	
Arsenic(d) (21-616 ug/g)	$S = 0.188X + 0.98$ $SR = 0.0857X + 2.95$		5A,6A,7A,7B
Selenium(d) (3.98-105 ug/g)	$S = 0.192X + 2.06$ $SR = 0.187X + 0.85$		All data sets corrected
Barium(e) (123-11630 ug/g)	No equation developed. See App. C, in main report, for individual statistics.		
Silver(e) (37-344 ug/g)	No equation developed. See App. C, in main report, for individual statistics.		

(a) Weighted least squares technique.
(b) Indicates the waste deleted to arrive at the regression equation presented.
(c) Solid waste data set that was corrected for errant data points. See Section 5, Results and Discussion for further details.
(d) Analyzed by graphite furnace atomic absorption (GFAA). All other analyses by flame atomic absorption (FLAA).
(e) The individual data sets were too variable to fit a linear relationship thus no equations were developed.

selenium, were 19.7% and 29.6% respectively while the single-analyst precision values, as %RSD-SR, were 11.4% and 23.0% respectively.

The seven matrices contained background concentrations of aluminum, calcium, iron, magnesium, manganese, potassium, and sodium and did not require fortification. This element group had a mean %RSD of 11.8% and a mean %RSD-SR of 5.8%. The seven matrices had no detectable concentrations of cobalt, molybdenum, thallium, and vanadium and required fortification. This element group had a mean %RSD of 8.4% and a mean %RSD-SR of 5.1%. The background concentrations of beryllium, cadmium, chromium, copper, lead, nickel, and zinc ranged from detectable to nondetectable. As a result, fortification was required of those matrices when the elements were too low or undetectable. This group of elements had a mean %RSD of 7.4% and a mean %RSD-SR of 5.2%. Based on these mean %RSDs, the overall and single-analyst precisions were very similar for all elements, whether naturally present or spiked into the solid matrices.

SECTION 3

DESCRIPTION OF STUDY

ANALYTICAL METHODOLOGY

SW-846 Method 3050 requires the digestion of a 1-2 gram solid waste sample in nitric acid and hydrogen peroxide then reflux with either nitric acid or hydrochloric acid. In this study, dilute hydrochloric acid was used as the final reflux acid for the flame atomic absorption (FLAA) analyses of aluminum, antimony, barium, beryllium, calcium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, silver, sodium, thallium, vanadium and zinc. Dilute nitric acid was used for the final reflux prior to graphite furnace atomic absorption (GFAA) analyses of arsenic and selenium. A previous interlaboratory method validation study (3) characterized the performance of Method 3050 coupled with analyses by Inductively Coupled Plasma-Optical Emission Spectroscopy.

Silver and antimony, included originally in the Method 3050 digestion procedure, were removed from the September 1986 revision of the method. However, the two elements were included in the Request For Proposal Statement of Work and they were kept in this interlaboratory study.

SELECTION OF PARTICIPATING LABORATORIES

Twenty-four commercial laboratories responded to an abstract in the Commerce Business Daily inviting participants for this method validation study. Their technical proposals were evaluated based upon laboratory experience and quality control practices. Laboratories whose proposals were acceptable were evaluated further in a preaward performance evaluation study. The participants selected for the formal study were the eight laboratories with acceptable proposals who performed best in the preaward study.

The Bionetics Corporation - Hampton, Virginia Laboratory
J.M. Montgomery Consulting Engineers, Inc.
Spotts, Stevens and McCoy, Inc.
ERCO/ENSECO, Inc.
Thermo Analytical, Inc.
Skinner and Sherman, Inc.
Science Applications, Inc.
PPB Environmental Laboratories, Inc.

SELECTION OF SOLID WASTE MATRICES

Fourteen solid waste matrices were screened for the 23 elements of interest by ICP analyses and seven solid wastes were selected that contained naturally occurring elements over a relatively wide concentration range. These were:

Solid Waste	Source
1	National Bureau of Standards River Sediment, SRM-1645
2	US EPA, Hazardous Soil QC Sample #1
3	Ethyl Corp., Electroplating Sludge
4	Marshall Space Flight Center (MSFC), Electroplating Sludge
5	US EPA, Shale I QC Sample WP 386
6	Varland Metal Service Inc., Electroplating Sludge
7	EPA Supplied Electroplating Sludge

The results obtained from the ICP analyses by QARD, EMSL-Cincinnati, for the seven selected solid waste matrices are presented in Table 2. Aluminum,

Table 2. ICP RESULTS OF THE BACKGROUND CONCENTRATIONS FOUND IN THE SEVEN SOLID MATRICES. (UG/G)

	SOLID SAMPLE NUMBER						
Element	1	2	3	4	5	6	7
Al	3,200	4,000	11,000	4,800	5,800	500	1,000
Sb	-	-	-	-	-	130	-
As	-	-	-	-	-	-	-
Ba	-	-	3,900	-	300	190	-
Be	19	38	198	144	4.8	96	80
Ca	15,000	43,000	40,000	27,000	80,000	33,000	7,000
Cd	16	-	24	99	-	-	-
Cr	28,000	120	65	8,000	-	52,000	52,000
Co	-	-	-	-	-	-	-
Cu	100	-	310	820	46	13,000	1,100
Fe	80,000	18,000	20,000	36,000	24,000	48,000	66,000
Pb	750	250	13,500	1,050	-	850	820
Mg	6,600	24,000	2,300	2,000	34,000	14,000	1,900
Mn	620	390	790	300	350	570	540
Mo	-	-	-	-	-	-	-
Ni	-	30	160	500	-	34,000	42,000
K	220	300	23	1,480	1,300	200	500
Se	-	-	-	-	-	-	-
Ag	-	-	-	-	-	-	20
Na	400	180	1,200	70,000	3,300	3,200	33,000
Tl	-	-	-	-	-	-	-
V	-	-	-	-	-	-	-
Zn	1,780	-	1,600	13,300	80	17,800	110,000

- Below detection limit.

calcium, iron, magnesium, manganese, potassium, and sodium were present in all seven solid wastes at a wide range of concentrations and did not require fortification with spiking solutions. Cobalt, molybdenum, thallium, selenium, barium, arsenic, silver, antimony, vanadium, beryllium, cadmium, chromium, copper, lead, nickel, and zinc either did not occur naturally or occurred at such low concentrations in the solid matrices that fortification was required.

DESCRIPTION OF THE STUDY

The design of the study is based upon Youden's original non-replicate design for collaborative evaluation of analytical methods (1). In this design, samples are prepared in pairs such that the analyte concentrations of the pairs vary between 5-20% of the mean of the pairs. Seven Youden sample pairs, one for each solid matrix, were prepared for each trace element to cover the optimum range of the method as specified in SW-846 or to cover the concentration range naturally occurring in the matrices.

Each solid matrix was then dispensed into 160 bottles, 80 of which received 1.0 g aliquots with the remaining 80 receiving 1.2 g aliquots. This provided a Youden concentration pair based on the total micrograms of each element extracted. The bottles were further divided into four groups of 20. The first group, designated Mix 1, received no spiking solution and was the only mix tested as a dry powder. This group was to be used for the analyses of aluminum, calcium, iron, magnesium, manganese, potassium, and sodium. The second group of bottles, designated Mix 2, was spiked with a 4.0 mL aliquot of solution into each bottle containing 1.0 g of dried solid waste and the bottles containing 1.2 g received a 5.0 mL aliquot of spiking solution to complete a Youden concentration pair. Mix 2 was to be used for the analysis of beryllium, cadmium, chromium, cobalt, copper, lead, molybdenum, nickel, antimony, thallium, vanadium, and zinc. The designated groups, Mix 3 and Mix 4, received 4.0 mL and 5.0 mL aliquots in 1.0 g and 1.2 g bottles, respectively. Mix 3 was to be used for the analysis of arsenic and selenium, while Mix 4 was for silver and barium.

Instructions for sample preparation, analyses of blanks and quality control samples, instrumental analyses, and data report forms are included in Appendix A. Each participant was required to run a quality control sample after every sixth sample to verify that the analytical system was in control. If not in control, the problem was to be corrected and the last six samples rerun.

VERIFICATION AND HOMOGENEITY ANALYSES

To assure that the spiked matrix samples submitted to the participating laboratories were prepared properly, independent analyses were performed on all sample groups by QARD analysts. These analyses confirmed the accuracy of the spiking solutions.

All solid wastes, except NBS River Sediment SRM 1645, were dried, ground, sieved and mixed in a cross-flow blender for use in this study. Statistical analyses of paired samples confirmed the homogeneity of each matrix.

SECTION 4

TREATMENT OF DATA

The overall and single-analyst precision of Method 3050 for 23 trace elements were determined using statistical techniques which followed ASTM Standard Practice D2777-77 (6). Statistical analyses were performed using computer programs hereafter referred to as IMVS (2). IMVS includes: tests for the rejection of individual outliers, summary statistics by concentration level for mean recovery, overall and single-analyst standard deviation (precision) and the linear relationship between standard deviation and mean recovery (7). A detailed description of the statistical analytical procedures is presented below.

REJECTION OF OUTLIERS

Spurious data points are always a part of any set of data collected during an interlaboratory test program. It is important to identify and remove these data points because they can result in values of summary statistics which are not representative of expected performance. However, some erratic data may be directly related to some facet of the method itself. Therefore, spurious data points should not be removed indiscriminately, and any points that are removed should be clearly identified since further investigation of the analytical conditions related to the outliers might be of value. Data rejected as outliers for this study, as a result of the test for outliers, have been identified by the symbol "*" in the raw data tables.

YOUDEN'S LABORATORY RANKING PROCEDURE

In some cases the analytical values reported by a specific laboratory are so consistently high or low that a large systematic error is attributed to that laboratory. These data are not representative of the method and should be rejected using Youden's ranking test (1). This procedure is normally applied to several sample pairs among the same matrix. However, since in this study the seven Youden pairs were prepared from seven different solid wastes its use was judged inappropriate and was not applied.

TESTS FOR INDIVIDUAL OUTLIERS

For the solid waste matrices, the data were grouped into 14 subsets defined by concentrations of elements in the seven Youden pairs tested in the study. For each subset of data, all missing, "less than", zero, and "nondetect" data were rejected. Next, the test for individual outliers of Thompson (5) suggested in the ASTM Standard Practice D2777-77 (6), was applied to the data at a 5% significance level. If a data point was rejected based on this test, it was removed from the subset, and the test was repeated using the remaining data in the subset. This process was continued until no additional data could be rejected.

STATISTICAL DATA SUMMARIES

Several summary statistics were calculated using the data remaining for each concentration level after rejection of outliers. These statistics include: the number of retained data points, the mean recovery, the absolute overall standard deviation, the percent relative overall standard deviation, the absolute single-analyst standard deviation, and the percent relative single-analyst standard deviation. The basic formulas used to calculate these statistics are presented below where X_1, X_2, . . ., X_n denote the values of the n retained data points for a specific concentration level.

Mean recovery ($\bar{X}$):

$$\bar{X} = \frac{1}{n} \sum_{i=1}^{n} X_i$$

The usual concentration notation for mean recovery is $\bar{X}$. However, the symbol X will be used in this report to be consistent with IMVS computer output.

Overall Standard Deviation (S):

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (X_i - X)^2}$$

and

Percent Relative Overall Standard Deviation:

$$\% \text{ RSD} = \frac{S}{X} (100)$$

The overall standard deviation (S) indicates the precision associated with measurements generated by a group of laboratories. This represents the broad variation in the data collected in an interlaboratory study. However, a measure of how well an individual analyst can expect to perform in his own laboratory is another important measure of precision. This single-analyst precision, denoted by SR, was estimated for each Youden pair by

$$SR = \sqrt{\frac{1}{2(m-1)} \sum_{i=1}^{m} (D_i - \bar{D})^2}$$

where

m = the number of complete sets of Youden pair observations remaining after outliers have been removed.

D_i = the difference between the observations in the ith Youden pair

and

$\bar{D}$ = the difference between the two averages of the D_i values.

The percent relative single-analyst standard deviation was calculated by

$$\% \text{ RSD-SR} = \frac{SR}{X^*}(100)$$

where X^* is the average of the two mean recovery statistics corresponding to the two concentration levels defining the particular Youden pair.

These summary statistics provide detailed information on the mean recovery and precision of the data obtained for each concentration level. One objective of the statistical analysis of the data is to summarize the information about mean recovery and precision which is contained in the statistics.

A systematic relationship exists between the precision statistics (S and SR) and the mean recovery (X). Usually, these systematic relationships can be adequately approximated by a linear relationship (i.e., by a straight line). Once these straight lines are established, they can be used to conveniently summarize the behavior of the method within a solid type, and they can aid in comparing the behavior of the method among solid types. In addition, they can be used to obtain estimates of the precision at any concentration level within the applicable range studied. They can also be used to predict the behavior of the method when used under similar conditions. These important relationships are discussed on the following pages.

STATEMENT OF METHOD PRECISION

The precision of the method is characterized by the relationships between precision statistics (S and SR) and mean recovery (X). In order to obtain a mathematical expression for these relationships, regression lines of the form

$$S = d + eX \qquad (1)$$

and

$$SR = f + gX^* \qquad (2)$$

were fitted to the data by regression techniques.

As discussed previously, the values of X and X* often vary over a wide range. In such cases the standard deviation statistics associated with the larger mean recovery values will dominate the regression lines. This will produce relatively larger errors in the estimates of S and SR at the lower mean recovery value. Therefore, a weighted least squares technique was also used to establish the values of the parameters d, e, f and g in Equations (1) and (2). The weighted least squares technique was performed by dividing both sides of Equation (1) by X resulting in Equation (3).

$$\frac{S}{X} = d\left(\frac{1}{X}\right) + e \qquad (3)$$

and dividing both sides of Equation (2) by X* resulting in Equation (4)

$$\frac{SR}{X^*} = f\left(\frac{1}{X^*}\right) + g \qquad (4)$$

The (S/X) values were regressed against the (1/X) values and the (SR/X*) values were regressed against the (1/X*) values using ordinary least squares to obtain estimates for the parameters d, e, f and g.

Equations (1) and (2) were obtained from Equations (3) and (4). The slope (d) for Equation (3) is the intercept (d) for Equation (1), and the intercept (e) for Equation (3) is the slope (e) for Equation (1). Similarly, the slope (f) for Equation (4) is the intercept (f) for Equation (2), and intercept (g) for Equation (4) is the slope (g) for Equation (2).

Given Equations (1) and (2), the percent relative overall standard deviation and the percent relative single-analyst standard deviation are;

$$\%RSD = \left(\frac{d}{X} + e\right)(100) \qquad (5)$$

and

$$\%RSD\text{-}SR = \left(\frac{f}{X^*} + g\right)(100) \qquad (6)$$

respectively. If the absolute value of the ratio (d/X) is small relative to the slope (e), then the percent relative overall standard deviation can be approximated by e(100) over the applicable range of mean recovery values. Similarly if the ratio (f/X*) is small relative to the slope (g), then the percent relative single-analyst standard deviation can be approximated by g(100) over the applicable range of mean recovery values. If the ratios (d/X) and (f/X*) are not small relative to the slopes (e) and (f), then the percent relative standard deviations depend upon the values of the mean recovery statistics X and X*, and they should be evaluated separately for each value of X and X*.

SECTION 5

RESULTS AND DISCUSSION

The objective of this study was to characterized the performance of SW-846 Method 3050 in terms of overall precision and single-analyst precision for 23 trace metals in sludges, soils and sediments. The IMVS computer programs were used to summarize the raw data and compile statistics for mean recovery, overall standard deviation, overall percent relative standard deviation, single-analyst standard deviation, and single-analyst percent relative standard deviation.

The study was intended to answer two major questions about method performance:

(1) Does Method 3050 digestion procedure work equally well on the solid wastes studied?

(2) Do the matrix effects encountered from any of the seven solid wastes studied prevent establishment of a linear relationship between precision and mean recovery?

The following decisions on data treatment were made on the statistics generated by the IMVS computer programs:

(1) Both overall and single-analyst precision regression equations were recalculated after removal of atypical solid waste data.

(2) Weighted linear regression equations were not calculated with less than five sets of acceptable solid waste data. In these situations

no regression equation was reported and it was concluded that the data for the element had a strong matrix dependence. The reader is referred to Appendix C, Statistical Summaries for the precision statistics for each solid waste.

(3) For comparison purposes, a concentration value of five times (5x) the low concentration limit of the range studied was used as the mean recovery, in the regression equations, to calculate %RSDs. This value is believed to be representative of the reported data in this study.

REJECTION OF OUTLIERS

Of the 2576 analytical results submitted for this study, 75 were rejected as outliers using Thompson's test for individual outliers (5) at the 5% significance level. A summary of the outliers by element and by laboratory is presented in Table 3. Rejected data are presented in Appendix B marked with an asterisk "*". Barium and selenium had the most outliers, 10 and 9 respectively; thallium and beryllium had none.

Thompson's test for individual outliers (5) is not rigorous enough to identify and remove two errant data points in a set of data that are very close or which balance each other at opposite ends of the data set. As a result, two or more data points in a data set can protect each other from rejection even though they are not representative of the set.

A typical example of such a situation is represented by the cadmium data illustrated below.

TABLE 3. NUMBER OF REJECTED DATA POINTS BY LABORATORY AND ELEMENT ACROSS ALL WASTES.

Element	Laboratory Code								
	001	002	003	004	005	006	007	008	Total
Al					1	2			3
Sb				7					7
Ba				2		8			10
Be									0
Ca				3	1				4
Cd	2		2			1		2	7
Cr				1			1	1	3
Co						1			1
Cu						3			3
Fe					1				1
Pb				1	3				4
Mg				1	1	1			3
Mn	5								5
Mo	1				1				2
Ni				1					1
K		2					1		3
Ag		1							1
Na	1				1	1	1		4
Tl									0
V			1						1
Zn					1	1			2
As						1			1
Se			2	1		6			9
Total	9	3	5	17	10	25	3	3	75

Lab No.	Reported value (ug/g)
1	7.4
2	7.9
3	7.7
4	14.6
5	11.8
6	7.3
7	8.9
8	6.4

Based on technical judgement, these errant data points were removed and new summary statistics calculated for the remaining solid waste data. The solid waste data set which was corrected is identified in the extreme right hand column in Table 1.

MEAN RECOVERY

It was not possible to calculate percent recovery statistics because true concentration values are not available in "natural" waste samples. Since the percent recovery is normally reported from the IMVS program, reference concentration values are reported in Appendix B, Raw Data, and Appendix C, Statistical Summaries. These reference values generated either from Neutron Activation Analyses (NAA) or, ICP analyses should not be used to characterize Method 3050.

OVERALL PRECISION

Overall standard deviation (S) value was computed for each solid waste to provided 14 estimates of precision (a pair for each solid waste) for each element. The overall percent relative standard deviation (%RSD) of the method is characterized by the relationship of the overall standard deviation (S) to the mean recovery (X) of each element among the seven solid wastes tested. In order to obtain a mathematical expression for this relationship, a weighted linear regression line was fitted to the data.

Since the concentrations of elements tested covered a wide range of values, the precision statistics at the higher concentrations tended to dominate the fitted regression line producing relatively larger errors in the estimates of precision at the lower concentrations. To reduce this problem, the weighted least squares technique was used. Chapter 4, Treatment of Data, describes the weighted least squares technique in further detail. The resultant weighted linear regression equation can be used to estimate the overall precision at any concentration of the element within the range studied. These equations are presented in Table 1.

Table 4 presents overall precision %RSD estimates, calculated from the equations presented in Table 1. Antimony and silver, which are no longer included in Method 3050, are listed in the tables but are not included in the following discussions.

The mean %RSD for all FLAA elements was 9.4%. Only two of these elements, aluminum (22.4%) and potassium (22.4%), had %RSDs greater than 15%, while fifteen elements had %RSDs less than 10%. The naturally occurring elements: aluminum, calcium, iron, magnesium, manganese, potassium and sodium, which did not require fortification, had a mean %RSD of 11.8%. Cobalt, molybdenum, thallium, and vanadium, which required spiking of all seven solid wastes, had a mean %RSD of 8.4%. Beryllium, cadmium, chromium, copper, lead, nickel, and zinc which were evaluated with a combination of spiked and unspiked solid wastes had a mean %RSD of 7.4%. Based on the mean %RSDs for these three groups, the overall precision obtained for all elements, was very similar whether naturally present or spiked into the solid matrices.

The %RSD for barium is not presented in Table 4 because the precision of barium in each of the seven waste samples was highly variable with no apparent relationship among the data points. Although background analyses were not performed for anions on the solid wastes, background levels of sulfur and sulfur oxides are suspected to have precipitated barium as a sulfate in the digestion procedure.

TABLE 4. OVERALL PRECISION, as %RSD, CALCULATED AT FIVE TIMES THE LOWEST LEVEL TESTED IN THIS STUDY

Element	Conc. Used	%RSD(a)
Aluminum	3575 ug/g	22.4%
Antimony(b)	315 ug/g	17.8%
Beryllium	27.5 ug/g	8.1%
Cadmium	28.0 ug/g	7.2%
Calcium	59500 ug/g	8.8%
Chromium	335 ug/g	9.4%
Cobalt	455 ug/g	6.8%
Copper	330 ug/g	6.4%
Iron	84500 ug/g	6.0%
Lead	3575 ug/g	5.6%
Magnesium	10250 ug/g	6.8%
Manganese	1435 ug/g	8.2%
Molybdenum	900 ug/g	7.0%
Nickel(c)	5000 ug/g	6.9%
Potassium	2240 ug/g	22.4%
Sodium	1385 ug/g	8.4%
Thallium	645 ug/g	6.8%
Vanadium	1670 ug/g	13.0%
Zinc	465 ug/g	8.6%
Arsenic(d)	105 ug/g	19.7%
Selenium(d)	19.9 ug/g	29.6%
Barium(e)	615 ug/g	---
Silver(b)(e)	185 ug/g	---

(a) Calculated using the regression equations in Table 1.
(b) No longer included in Method 3050.
(c) The concentration range for nickel was so wide that use of 5x the lowest value would not have been a good measure of the %RSD.
(d) These elements were analyzed by graphite furnace atomic absorption (GFAA).
(e) No regression equations were developed. Three or more solid waste data pairs were deleted.

The %RSD for the GFAA elements, arsenic and selenium, were 19.7% and 29.6% respectively.

The individual overall precision versus mean recovery regressions are presented in the following plots. In all cases, a weighted linear regression equation was fitted first to the reported data for the seven solid waste Youden pairs. If one or more solid waste data pairs were not representative of the individual summary statistics in Appendix C, the solid waste(s) data pairs were removed and the weighed linear regression equation was replotted. The removal of solid waste(s) data pairs, therefore, suggests some degree of matrix interference with the element. Replotted regression equations are traced as solid lines and the deleted solid waste data pairs are labelled with their solid waste number. If both weighted linear regression equations are plotted, the equation most representative of the linear relationship is marked with an asterisk "*". The equations presented in Table 1 are the equations labelled with the asterisk. Those data points which fall on the outer lines of the plot represent points which exceed the scale of the plot.

Figure 1 through 14 are weighted linear regression plots for the elements: aluminum, beryllium, copper, iron, magnesium, manganese, nickel, potassium, sodium, thallium, vanadium, zinc, arsenic and selenium. These plots show the linear relationship between the overall precision and mean recovery for all seven solid waste pairs. In some instances the higher variability (poorer precision) of a solid waste pair may be offset by the lower variability (better precision) of another solid waste pair but the general trend of a linear relationship is present and does not change if the opposing solid waste pairs are deleted. However, it still suggests that these elements are exhibiting a matrix effect.

Figures 15 through 23 are weighted linear regression plots for the elements: antimony, cadmium, calcium, chromium, cobalt, lead, and molybdenum. A stronger matrix dependence was observed for these elements as compared to the elements in Figures 1-14. In each case one or two sample pairs were removed from the original plot because they varied from the rest of the data. The statistical summary section, Appendix C, was used initially to compare the %RSDs for each of the 14 individual data sets for the 7 solid waste pairs and to screen for atypical data pairs. A typical example is seen with antimony, in Table 33. In this table the overall %RSD ranges from 10% to 38% for solid wastes 2 through 6, while the %RSD varied from 44% and 59% for solid waste 1A and 1B to 4.6% and 6.4% for solid waste 7A and 7B. These latter matrices differ dramatically in concentrations from the other five matrices as illustrated in Figure 15. The solid line in Figure 16 was calculated with solid waste data 1 and 7 removed and better represents the linear relationship of the retained data and is therefore favored. Several elements will have more than one figure due to removal of one or more solid waste data pairs. The weighted linear regression equations are included at the bottom of each figure with a brief description of which solid waste data pairs were removed.

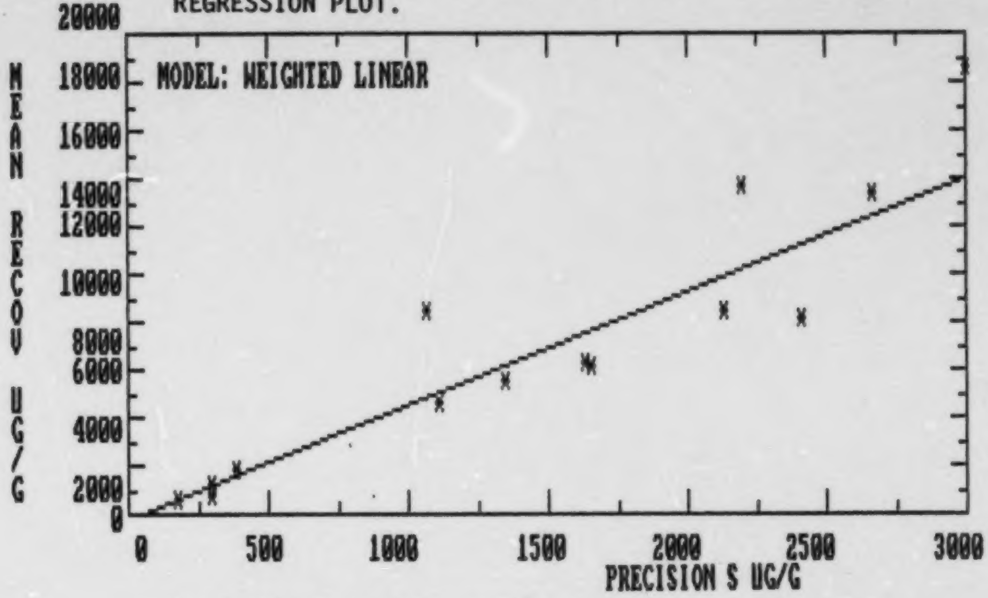
Barium statistics required the removal of three or more solid waste data pairs in order for the remaining data to approach a linear relationship. As discussed previously, it was assumed that a strong matrix dependence was suggested by the removal of three or more solid waste data pairs and that no weighted linear regression equation would be reported. The high variability observed among the barium solid waste pairs suggests the presence of sulfur or sulfur oxides in the solid wastes.

SINGLE-ANALYST PRECISION

The method validation study for 3050 was designed to measure both overall and single-analyst precision by providing samples as Youden sample pairs which can be used to evaluate statistically the within-laboratory precision (SR) and the between-laboratory precision (S).

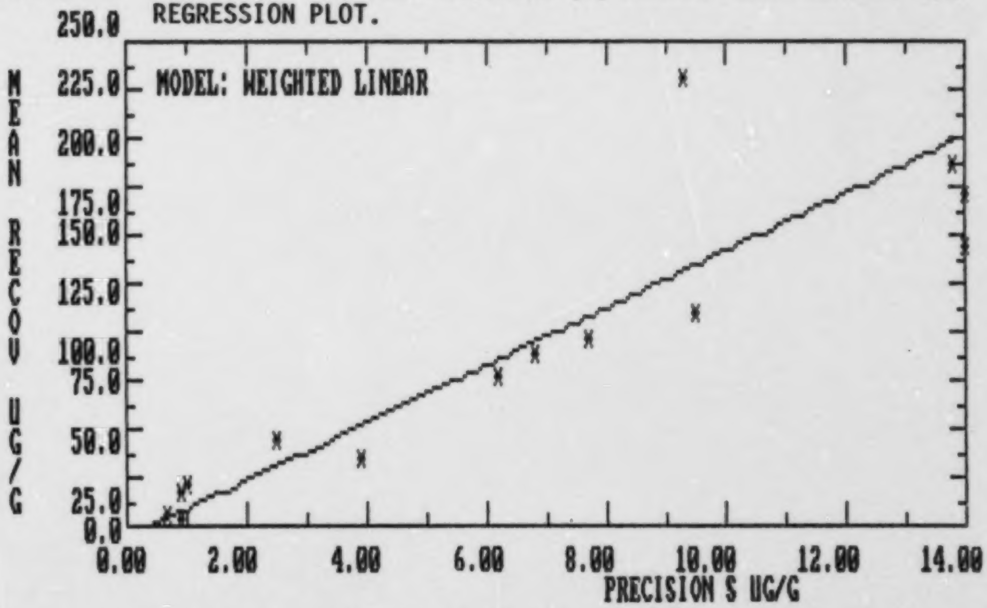
The IMVS computer programs calculated the single-analyst precision for each of the seven Youden concentration pairs for this study. These values can be found in Appendix C, Statistical Summaries. All seven data points,

FIGURE 1. ALUMINUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



S = 0.210X + 50.98* Includes all seven solid wastes.

FIGURE 2. BERYLLIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.

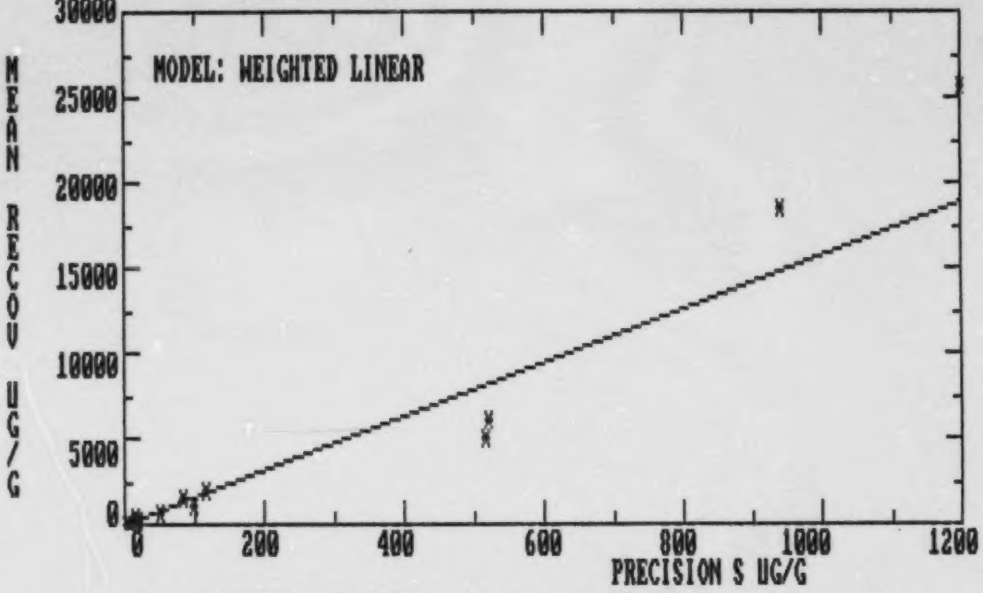


S = 0.0676X + 0.366* Includes all seven solid wastes.

---

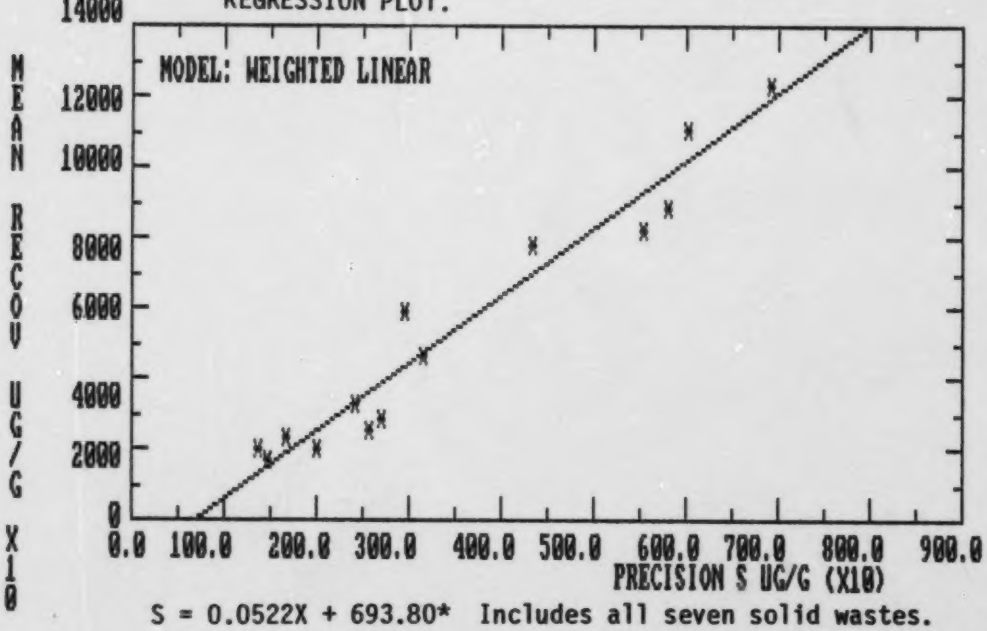
* Denotes the equation reported in Table 1.

FIGURE 3. COPPER: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



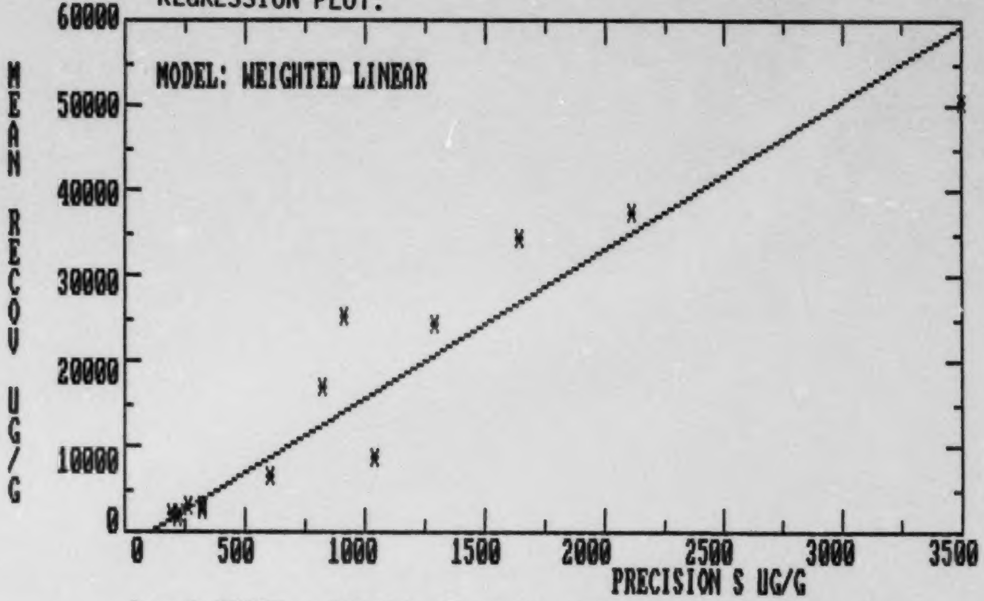
S = 0.0636X - 0.0031* Includes all seven solid wastes.

FIGURE 4. IRON: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



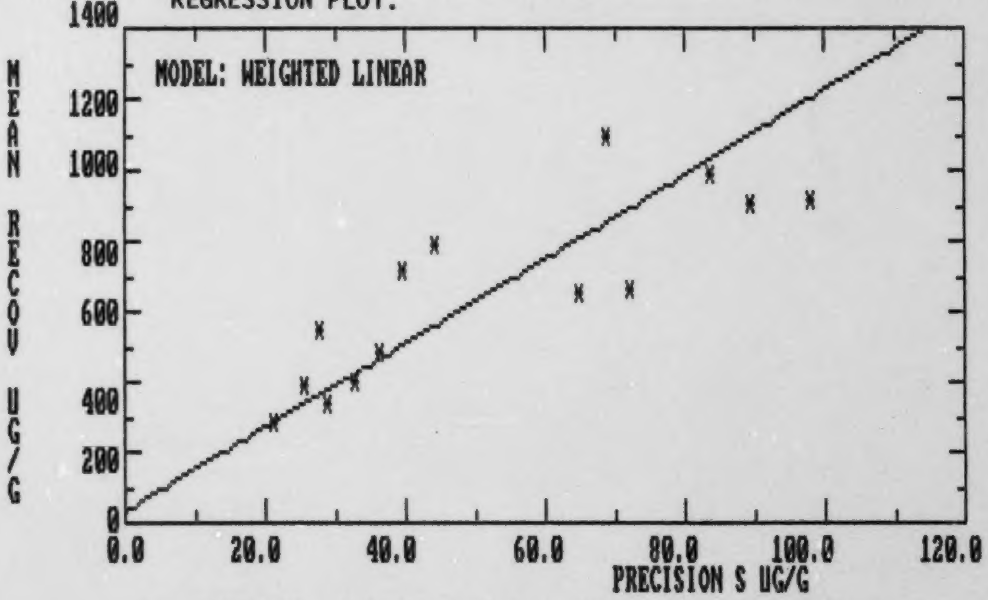
S = 0.0522X + 693.80* Includes all seven solid wastes.

FIGURE 5. MAGNESIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



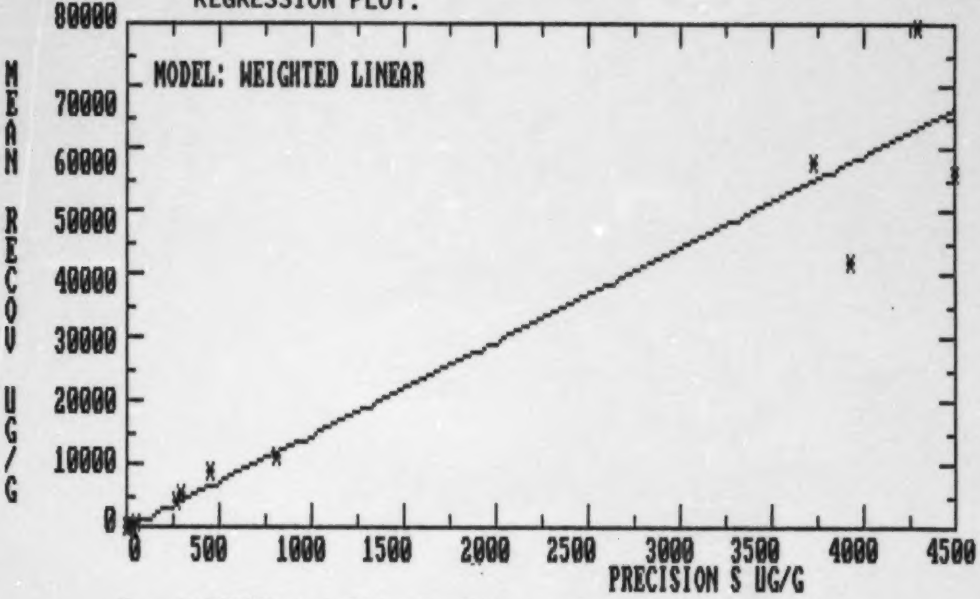
S = 0.0572X + 106.8* Includes all seven solid wastes.

FIGURE 6. MANGANESE: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



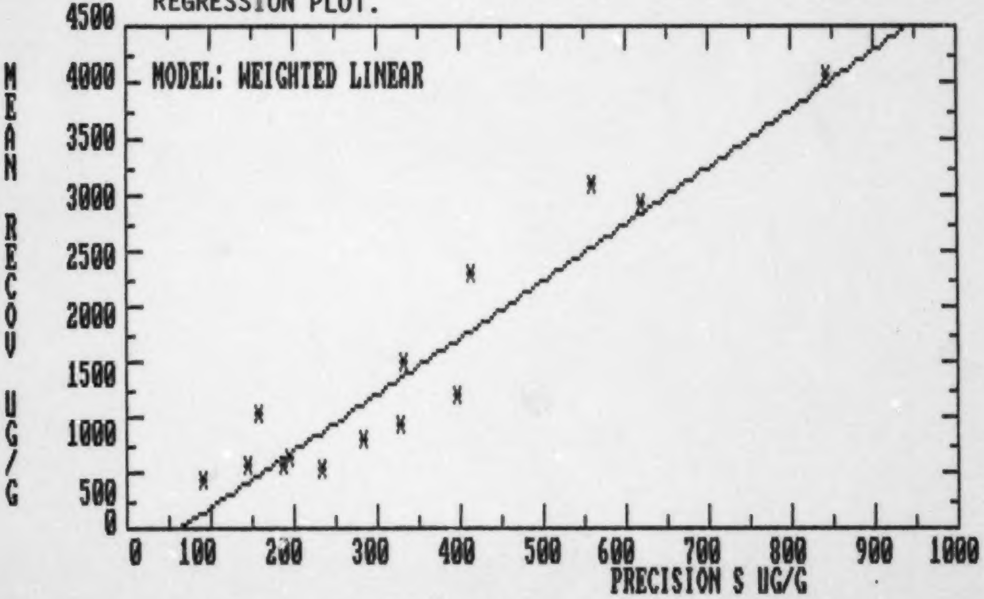
S = 0.0840X - 3.03* Includes all seven solid wastes.

FIGURE 7. NICKEL: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



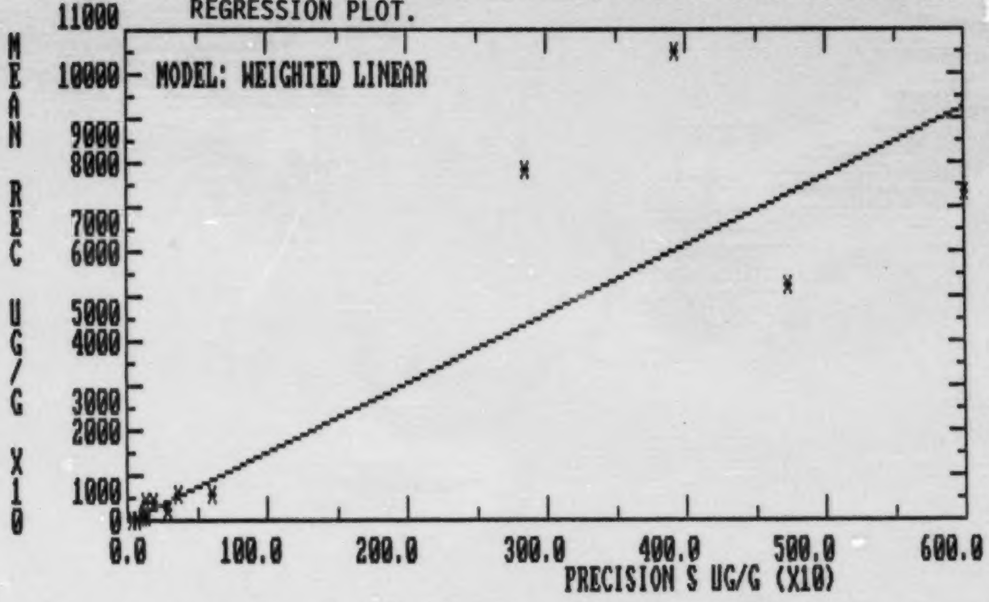
S = 0.0678X + 6.44* Includes all seven solid wastes.

FIGURE 8. POTASSIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



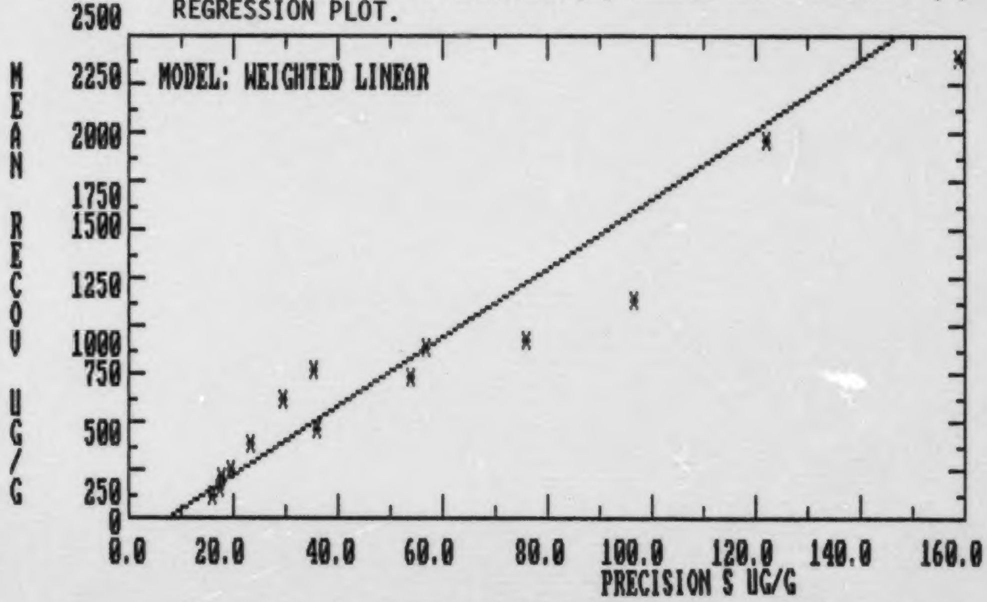
S = 0.196X + 61.84* Includes all seven solid wastes.

FIGURE 9. SODIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



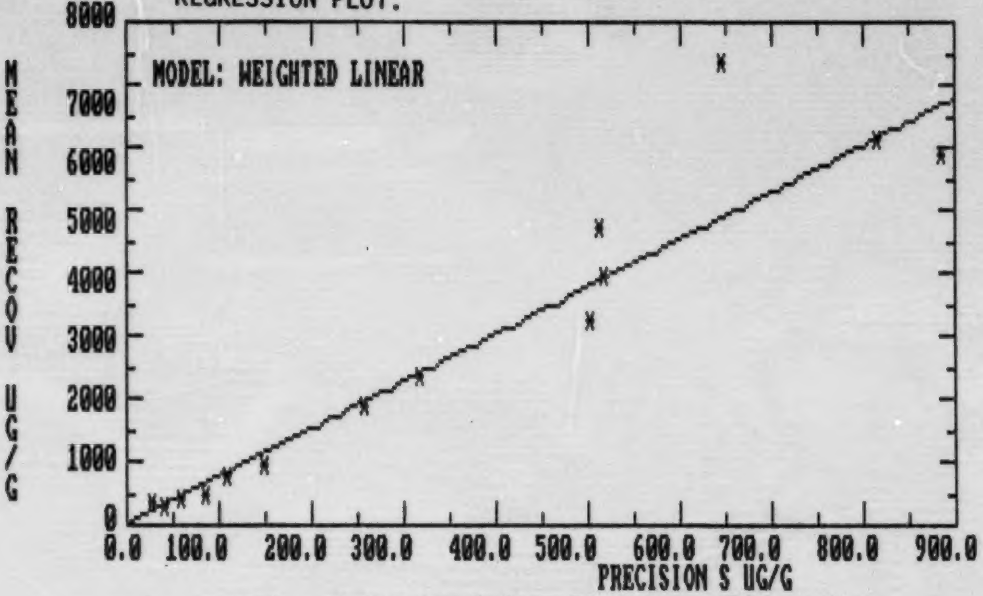
S = 0.0646X + 26.36* Includes all seven solid wastes.

FIGURE 10. THALLIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



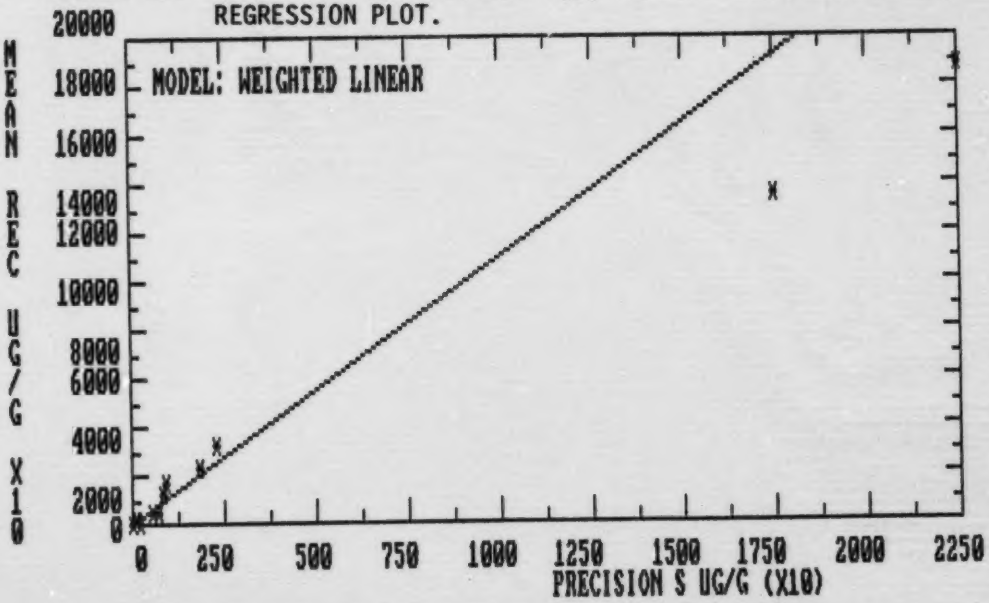
S = 0.0559X + 7.53* Includes all seven solid wastes.

FIGURE 11. VANADIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



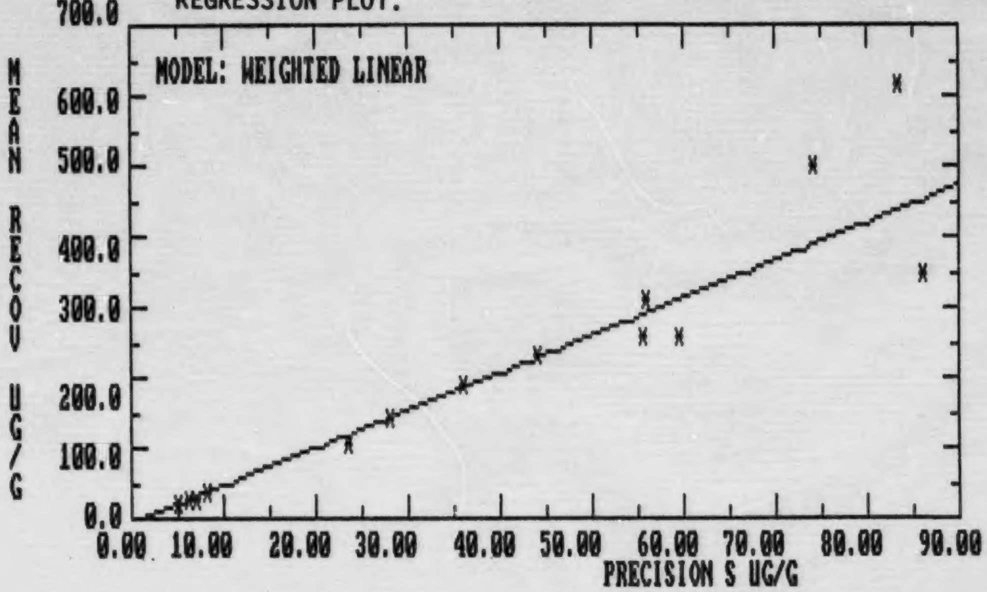
S = 0.133X - 4.24* Includes all seven solid wastes.

FIGURE 12. ZINC: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



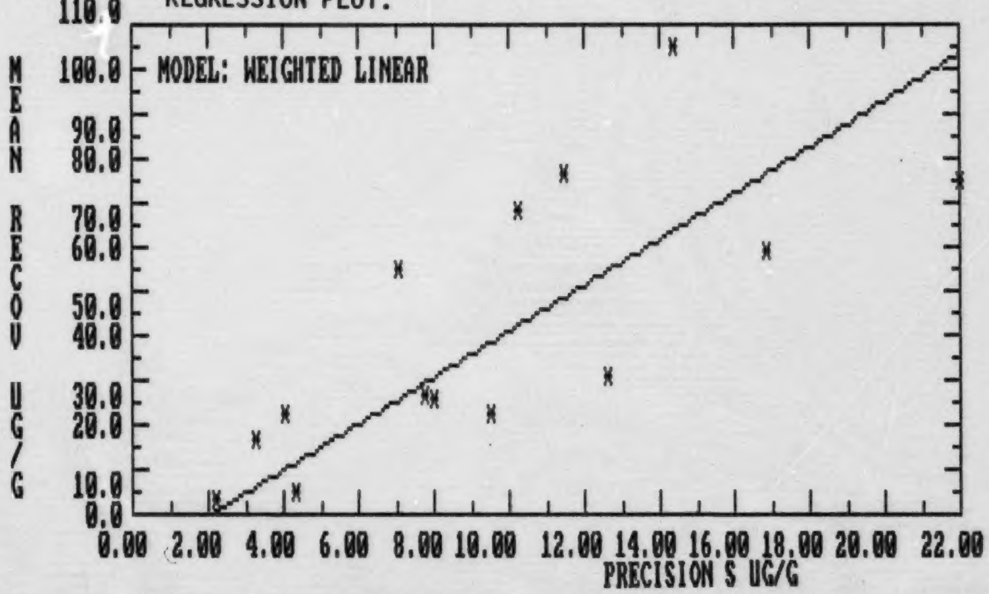
S = 0.0912X + 0.136* Includes all seven solid wastes.

FIGURE 13. ARSENIC: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



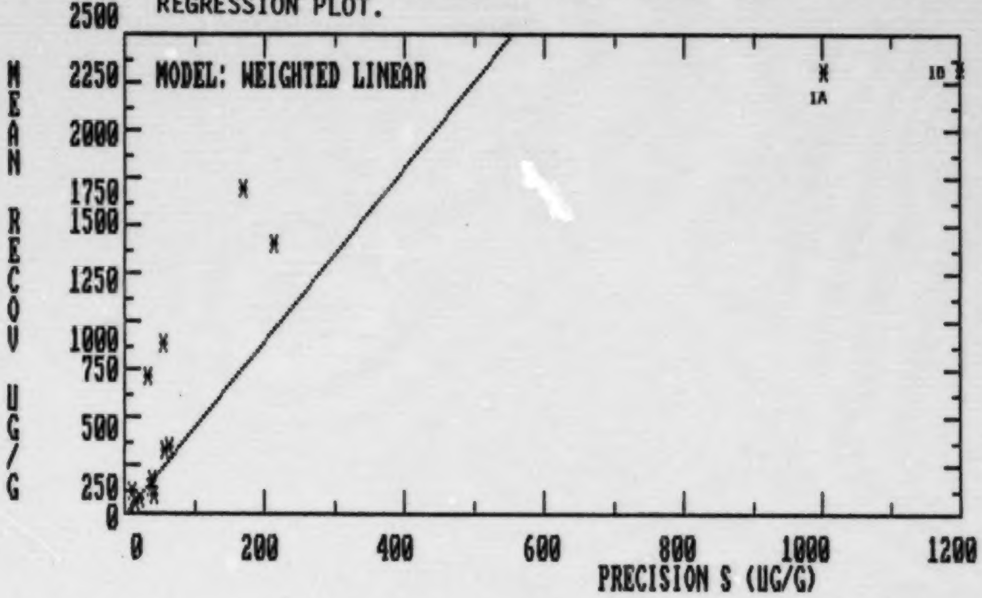
S = 0.188X + 0.975* Includes all seven solid wastes.

FIGURE 14. SELENIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



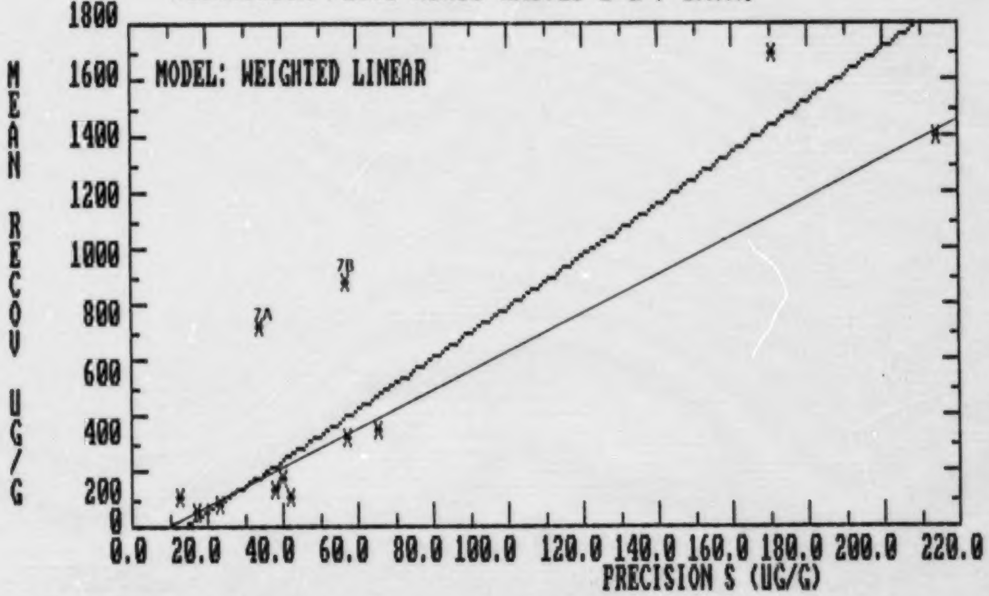
S = 0.192X + 2.06* Includes all seven solid wastes.

FIGURE 15. ANTIMONY: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



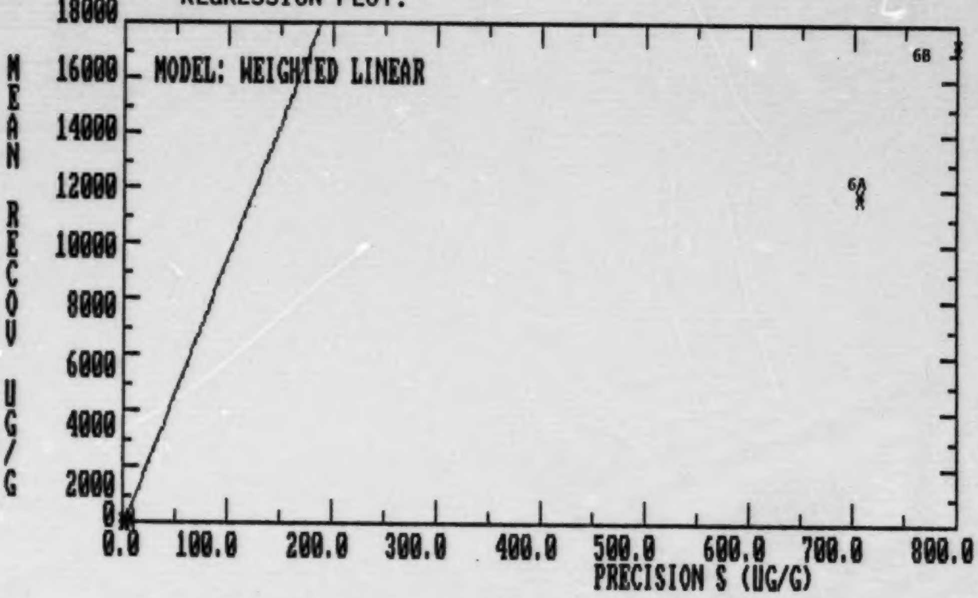
S = 0.221X + 2.48 Includes all seven solid wastes.

FIGURE 16. ANTIMONY: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 1 & 7 DATA.



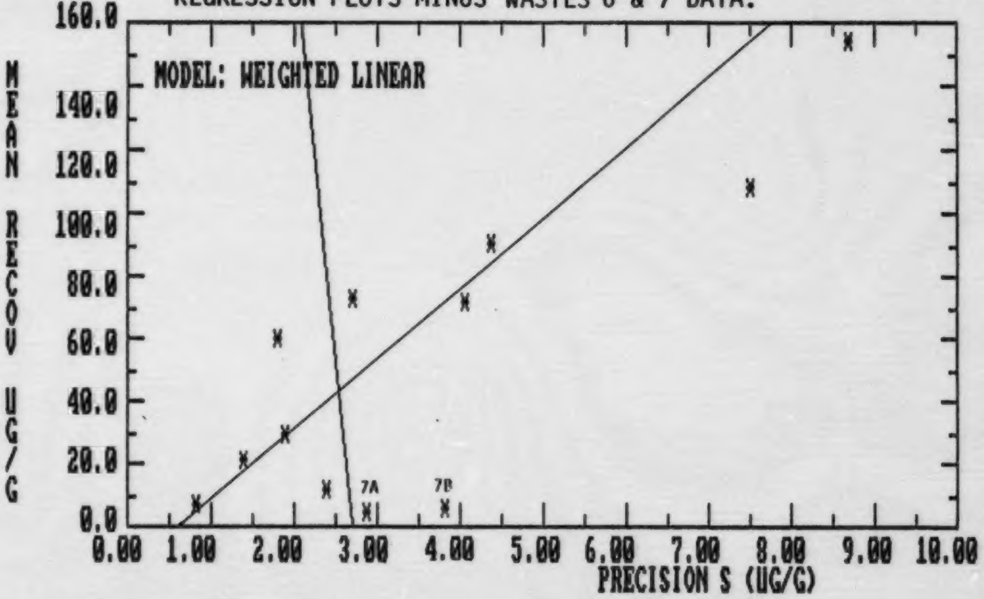
(---) S = 0.109X + 13.99 Solid Waste 1 removed.
(—) S = 0.145X + 10.58* Solid Wastes 1,7 removed.

FIGURE 17. CADMIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



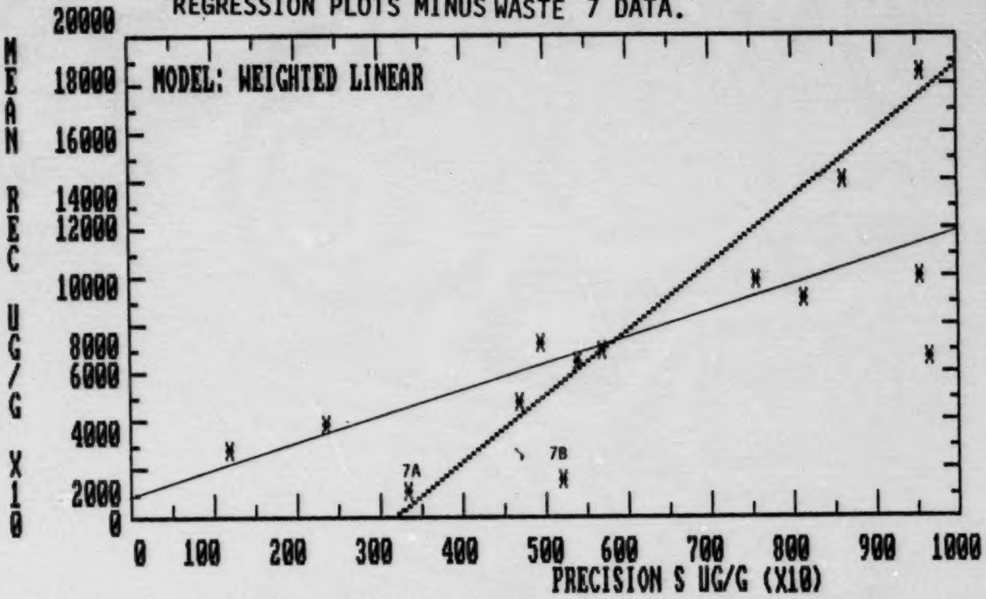
S = 0.0104X + 2.60 Includes all seven solid wastes.

FIGURE 18. CADMIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 6 & 7 DATA.



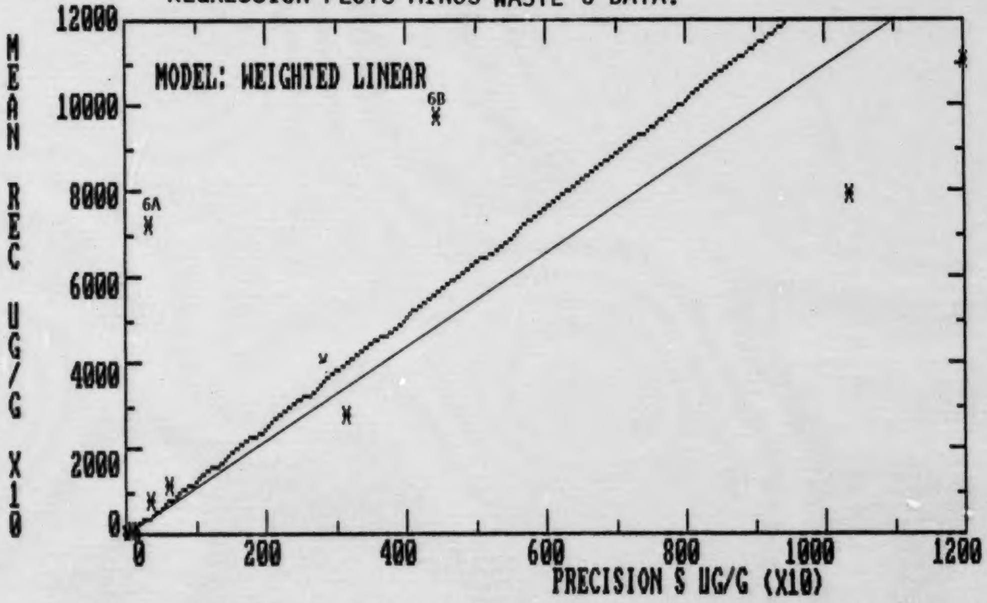
(---) S = -0.0039X + 2.72 Solid Waste 6 removed.
(——) S = 0.0437X + 0.782* Solid Wastes 6,7 removed.

FIGURE 19. CALCIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 7 DATA.



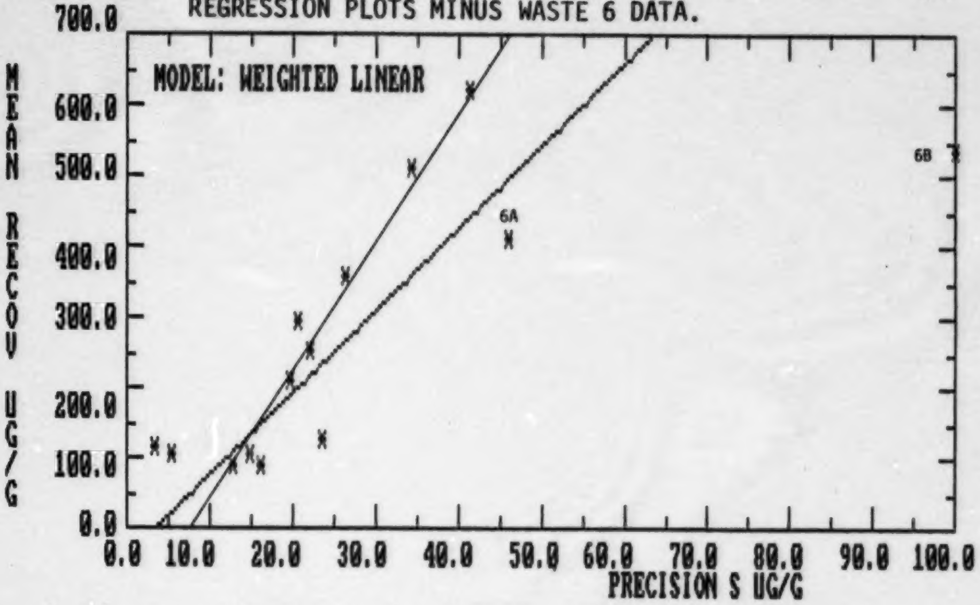
(---) S = 0.0364X + 3181 Includes all seven solid wastes.
(——) S = 0.0893X - 66.34* Solid Waste 7 removed.

FIGURE 20. CHROMIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 6 DATA.



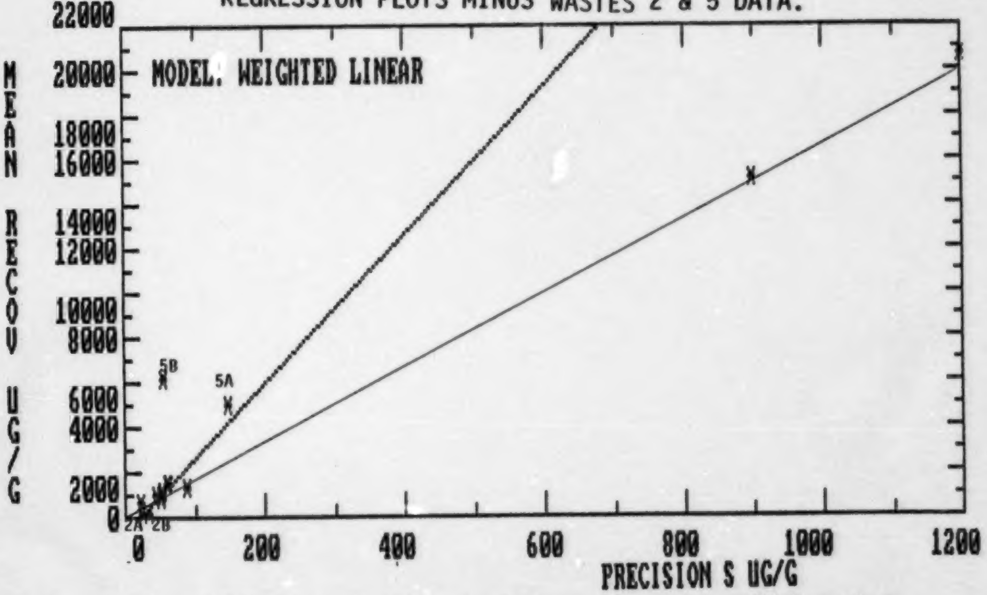
(---) S = 0.0790X + 8.55 Includes all seven solid wastes.
(——) S = 0.0923X + 0.73* Solid Waste 6 removed.

FIGURE 21. COBALT: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 6 DATA.



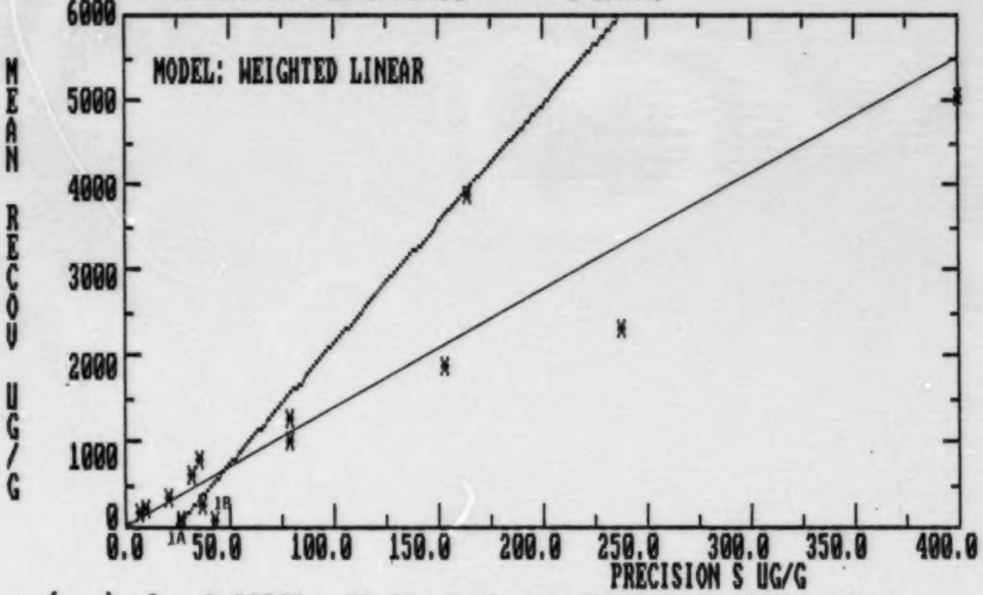
(---) S = 0.0858X + 3.46 Includes all seven solid wastes.
(—) S = 0.0525X + 7.22* Solid Waste 6 deleted.

FIGURE 22. LEAD: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 2 & 5 DATA.



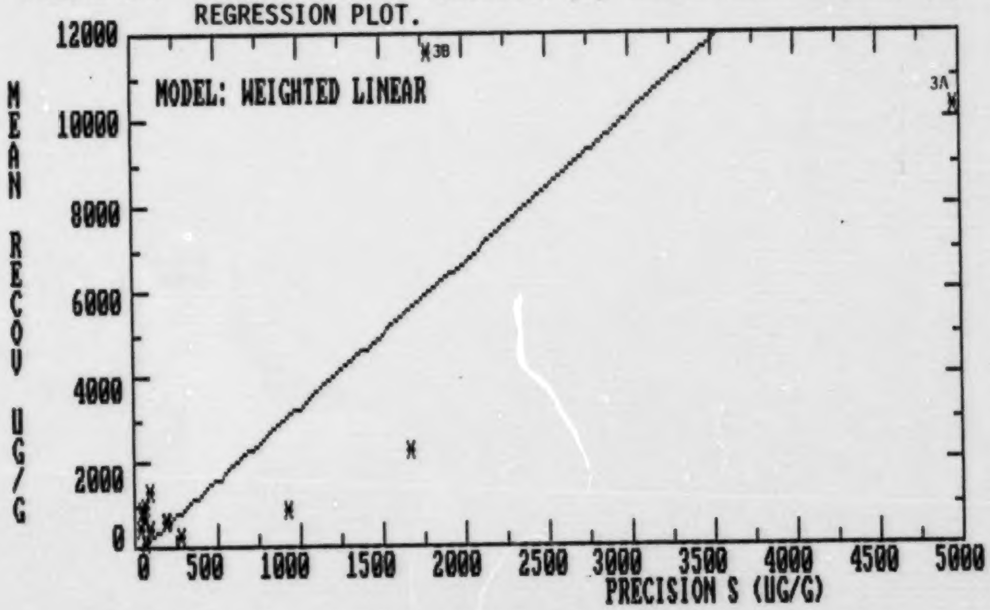
(---) S = 0.0300X + 21.93 Includes all seven solid wastes.
(—) S = 0.0607X - 17.42* Solid Wastes 2,5 removed.

FIGURE 23. MOLYBDENUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 1 DATA.



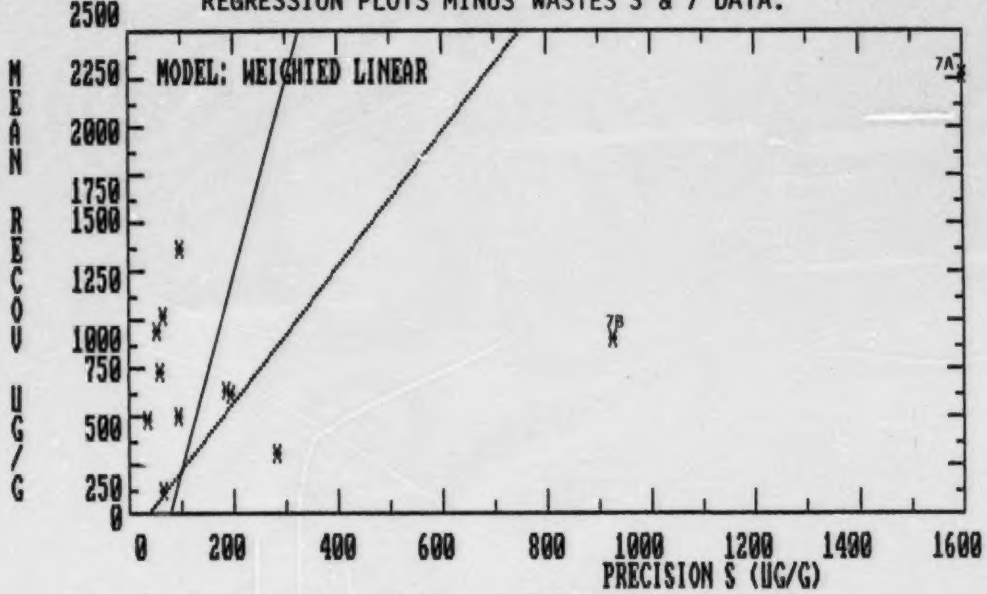
(---) S = 0.0356X + 23.40 Includes all seven solid wastes.
(——) S = 0.0739X - 3.50* Solid Waste 1 removed.

FIGURE 24. BARIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



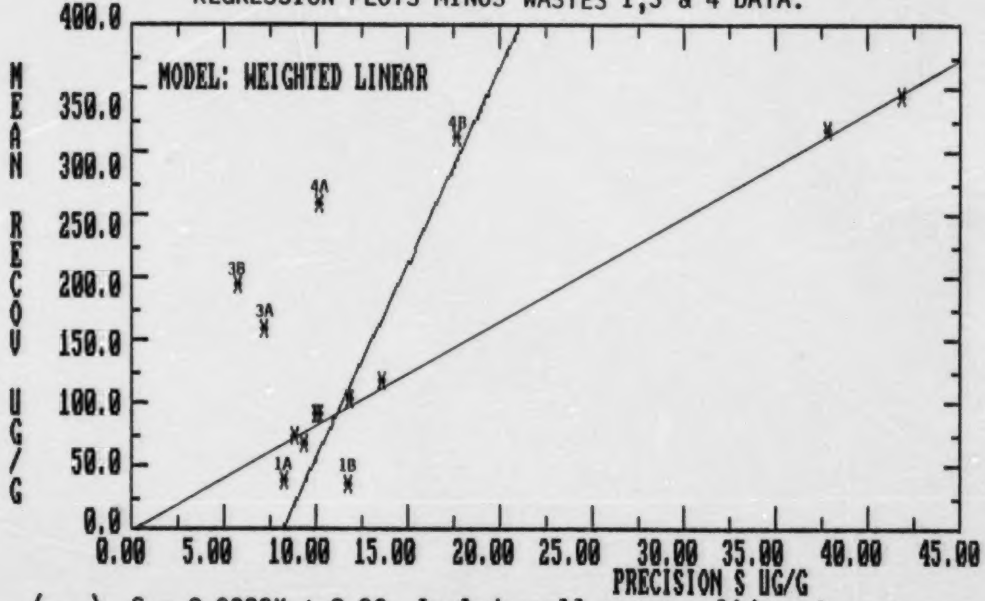
S = 0.292X + 35.75 Includes all seven solid wastes.

FIGURE 25. BARIUM: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 3 & 7 DATA.



(---) S = 0.285X + 37.48 Solid Waste 3 removed.
(——) S = 0.0898X + 74.62 Solid Wastes 3,7 removed.
No equation is favored-data is too variable.

FIGURE 26. SILVER: OVERALL PRECISION (S) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 1,3 & 4 DATA.



(---) S = 0.0322X + 8.23 Includes all seven solid wastes.
(——) S = 0.114X + 0.40 Solid Wastes 1,3,4 removed.
No equation is favored-three solid waste data points removed.

for each element, were reduced to a single linear regression equation.

A weighted least squares technique was used to fit the single-analyst precision data to the average of the two mean recoveries of the Youden pair. The weighted linear regression equations were selected, which best represents the single-analyst precision data, are presented in Table 1.

Table 5 presents the single-analyst precision %RSD-SR estimates calculated from the equations presented in Table 1. Antimony and silver, which are no longer included in Method 3050, are listed in the table but are not included in the discussions.

The mean %RSD-SR for all the FLAA elements was 5.4% and ranged from cobalt (4.2%) to aluminum (8.6%) The ratio of the mean overall precision, %RSD, to the mean single-analyst precision, %RSD-SR, was 1.7:1. The naturally occurring elements: aluminum, calcium, iron, magnesium, manganese, potassium and sodium had a mean %RSD-SR of 5.8%. Cobalt, molybdenum, thallium and vanadium which required spiking of all seven solid wastes, had a mean %RSD-SR of 5.1%. Beryllium, cadmium, chromium, copper, lead, nickel, and zinc were analyzed from a combination of spiked and unspiked solid wastes and had a mean %RSD-SR of 5.2%. Based on the mean %RSD-SRs for these three groups, the within-laboratory precision obtained for all elements, whether naturally present or spiked into the solid matrices, was very similar.

The GFAA elements, arsenic and selenium, had %RSD-SRs of 11.4% and 23.0% respectively and were the only elements with %RSD-SRs above 10%.

Barium %RSD-SR was not presented in Table 5 because at least three solid waste data sets would have had to be deleted in order that the retained data fit a linear relationship.

Plots of the single-analyst precision (SR) versus the average of the two mean recovery statistics for the Youden pair are presented in the following figures. In all cases, a weighted linear regression equation was fitted

TABLE 5. SINGLE-ANALYST PRECISION %RSD-SR CALCULATED AT FIVE TIMES THE LOWEST LEVEL TESTED IN THIS STUDY.

Element	Conc. Used	%RSD-SR(a)
Aluminum	3575 ug/g	8.6%
Antimony(b)	315 ug/g	8.3%
Beryllium	27.5 ug/g	5.0%
Cadmium	28.0 ug/g	5.9%
Calcium	59500 ug/g	5.1%
Chromium	335 ug/g	5.8%
Cobalt	455 ug/g	4.2%
Copper	330 ug/g	5.2%
Iron	84500 ug/g	3.8%
Lead	3575 ug/g	3.0%
Magnesium	10250 ug/g	4.3%
Manganese	1435 ug/g	5.7%
Molybdenum	900 ug/g	8.2%
Nickel(c)	5000 ug/g	4.5%
Potassium	2240 ug/g	8.1%
Sodium	1385 ug/g	5.4%
Thallium	645 ug/g	2.8%
Vanadium	1670 ug/g	5.2%
Zinc	465 ug/g	6.7%
Arsenic(d)	105 ug/g	11.4%
Selenium(d)	19.9 ug/g	23.0%
Barium(e)	615 ug/g	---
Silver(b)(e)	185 ug/g	---

(a) Calculated using regression equations in Table 1.

(b) No longer included in Method 3050.

(c) The concentration range for nickel was so wide that using 5x the lowest value would not have been a good measure of the %RSD.

(d) These elements were analyzed by graphite furnace atomic absorption (GFAA).

(e) No regression equations were developed. Three or more solid waste data pairs were deleted.

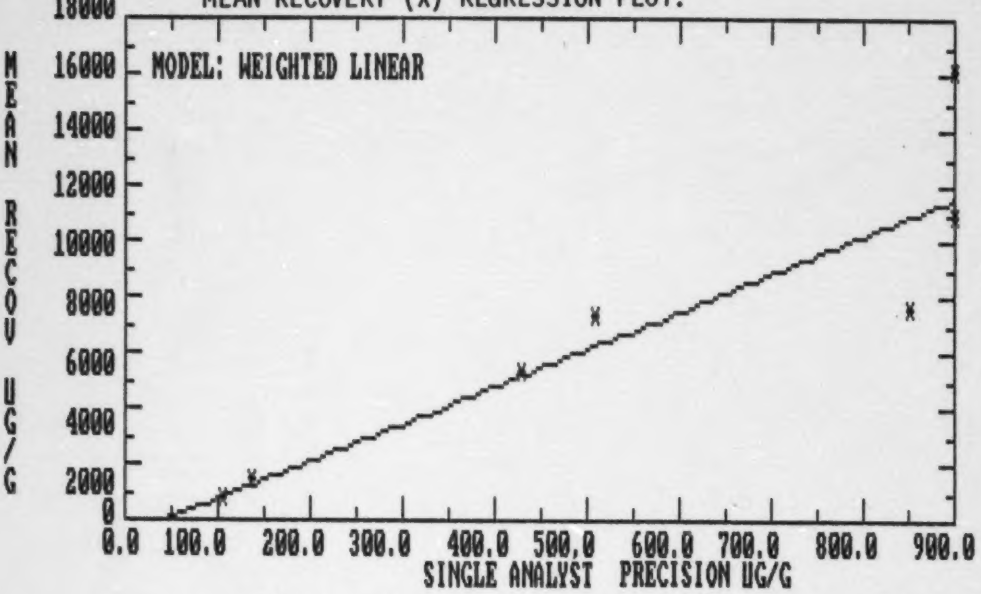
first to the reported data for the seven solid waste Youden pairs. If a solid waste(s) data pair were removed as not representative in the overall precision linear regression equation calculations, then those same solid waste(s) data were removed and the single-analyst linear regression equation recalculated. The removal of a solid waste data point suggests some degree of matrix effect on that element and the reader is directed to the Statistical Summaries, Appendix C, for statistics on the individual solid wastes in question. If a linear regression equation was replotted after deletion of solid waste data points the revised equation was traced as a solid line and the deleted solid waste data are labelled with the solid waste number. If two plots are present on the same figure both weighted linear regression equations are listed and the equation most representative of a linear relationship is marked with an asterisk "*". The favored single-analyst linear regression equations are those presented in Table 1.

Figures 27 through 40 are weighted linear regression plots for aluminum, beryllium, copper, iron, magnesium, manganese, nickel, potassium, sodium, thallium, vanadium, zinc, arsenic, and selenium. The plots for these fourteen elements suggest a linear relationship between the single-analyst precision data and the average of the two mean recoveries of the Youden pair for all seven solid matrices. In some instances the higher variability (poorer precision) of a solid waste data point may be offset by the lower variability (better precision) of another solid waste data point but the general trend of a linear relationship is still present and will not change if the opposing solid waste data points are deleted. The variability suggests a certain degree of matrix interference.

Figures 41 through 48 are weighted linear regression plots for antimony, cadmium, calcium, chromium, cobalt, lead, and molybdenum. A stronger matrix effect was observed for these elements as opposed to the previous element group. Observations made of the single-analyst precision plots show that deletion of the same solid waste data as was deleted in the overall precision calculations, did improve the fit of the retained data to a linear relationship. The dashed line represents the linear regression equation for all seven data points while the solid line represents the recalculated linear regression equation with the appropriate solid waste data removed. The deleted data are labelled with solid waste numbers. The original and recalculated linear regression equations are listed at the bottom of each plot with the recalculated equations being favored as more representative of the data.

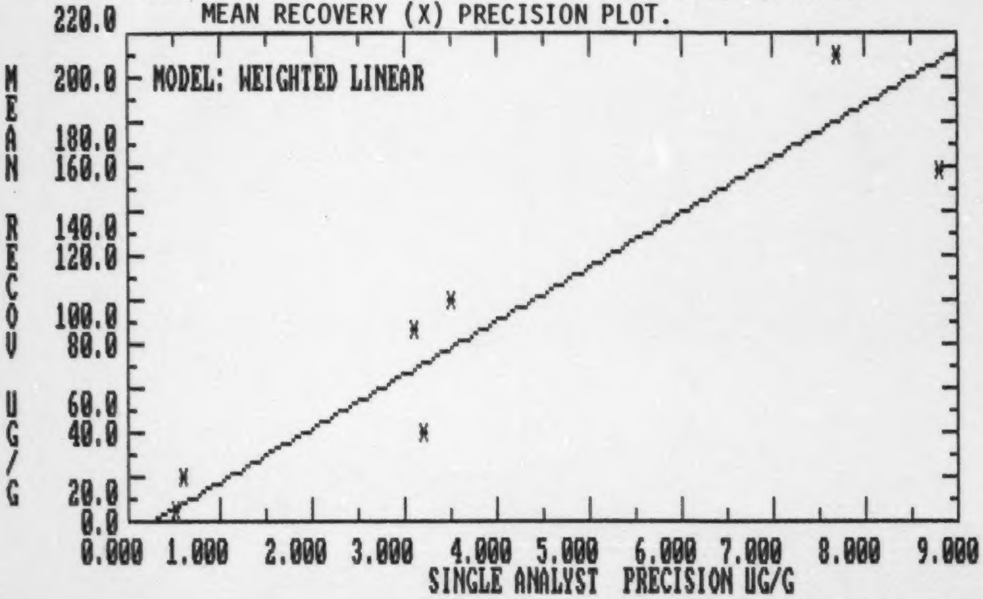
For barium, three or more data points had to be removed before the linear regression equations began to fit the data. A strong matrix dependence is suggested by the removal of three or more data points and as a result, no weighted linear regression equation was reported in Table 1. A possible explanation for the high degree of variability of barium data was discussed in the Overall Precision Section.

FIGURE 27. ALUMINUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



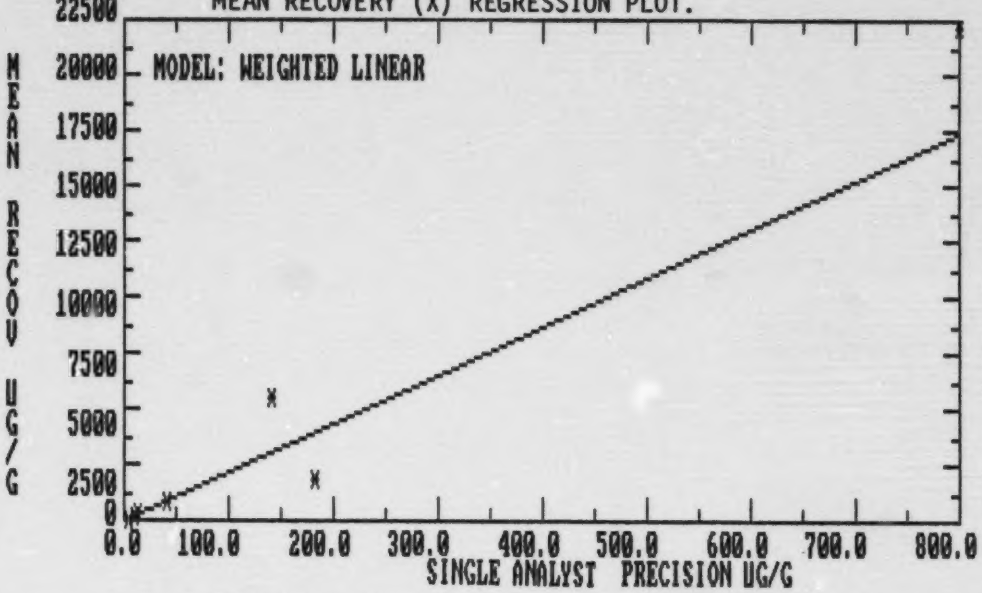
SR = 0.0742X + 41.17* Includes all seven solid wastes.

FIGURE 28. BERYLLIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) PRECISION PLOT.



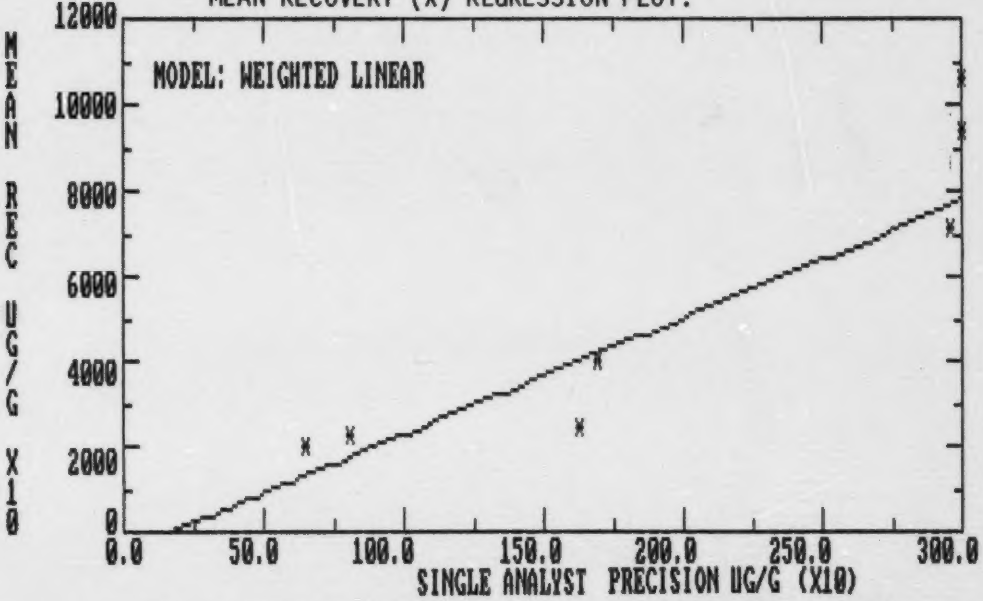
SR = 0.0411X = 0.26* Includes all seven solid wastes.

FIGURE 29. COPPER: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



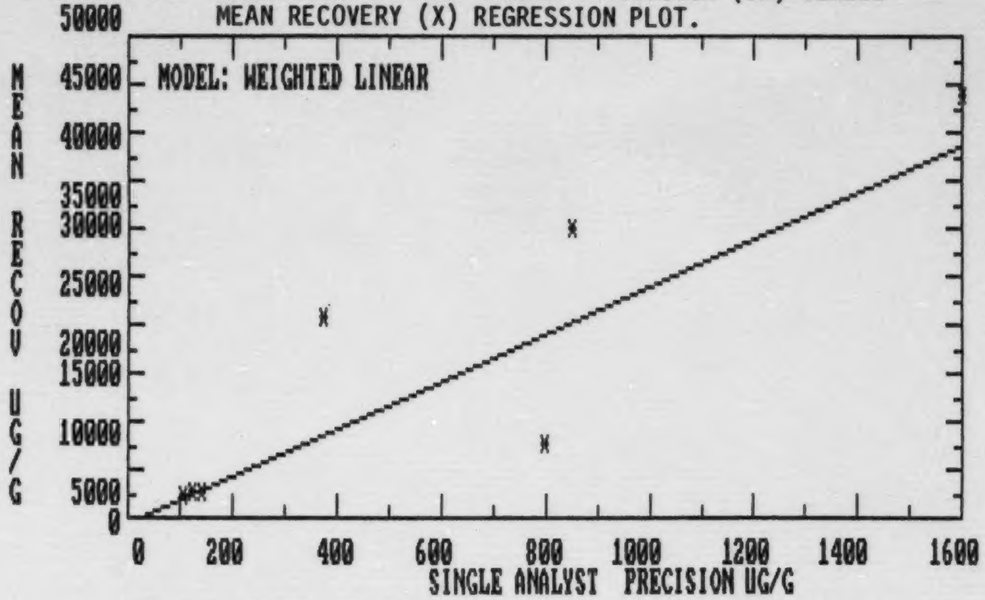
SR = 0.046X + 1.96* Includes all seven solid wastes.

FIGURE 30. IRON: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



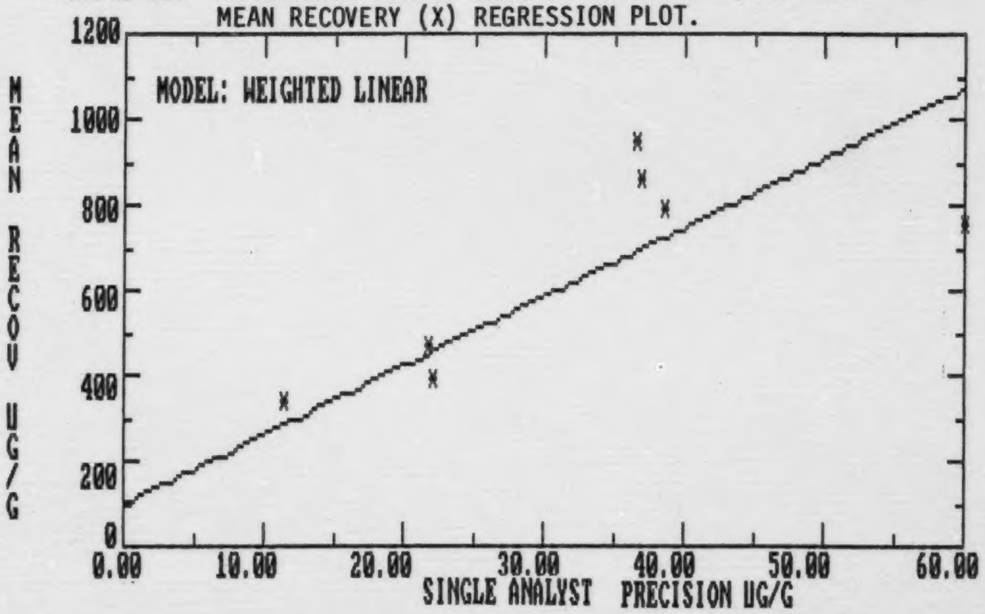
SR = 0.0364X + 162.0* Includes all seven solid wastes.

FIGURE 31. MAGNESIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



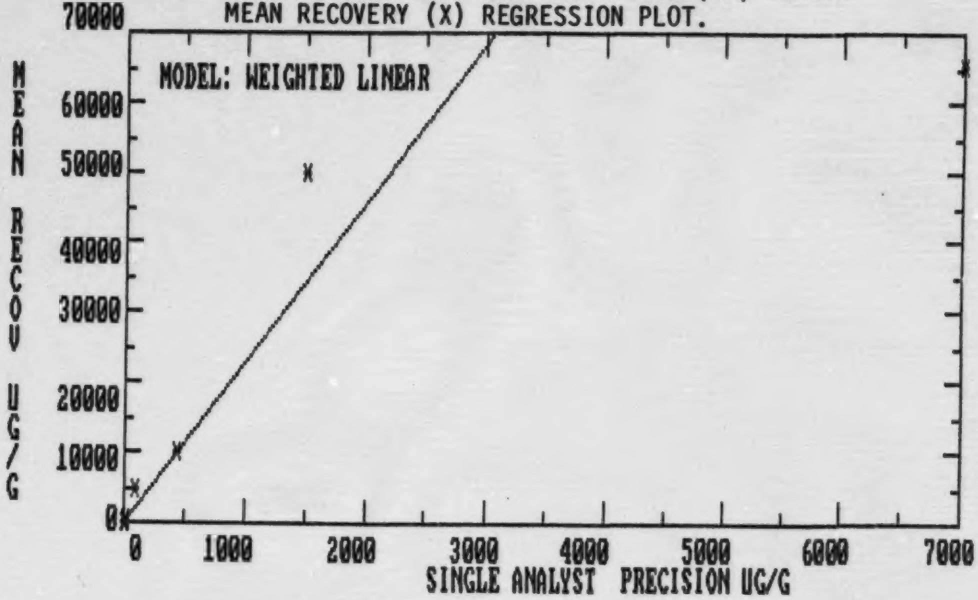
SR = 0.0408X + 26.44* Includes all seven solid wastes.

FIGURE 32. MANGANESE: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



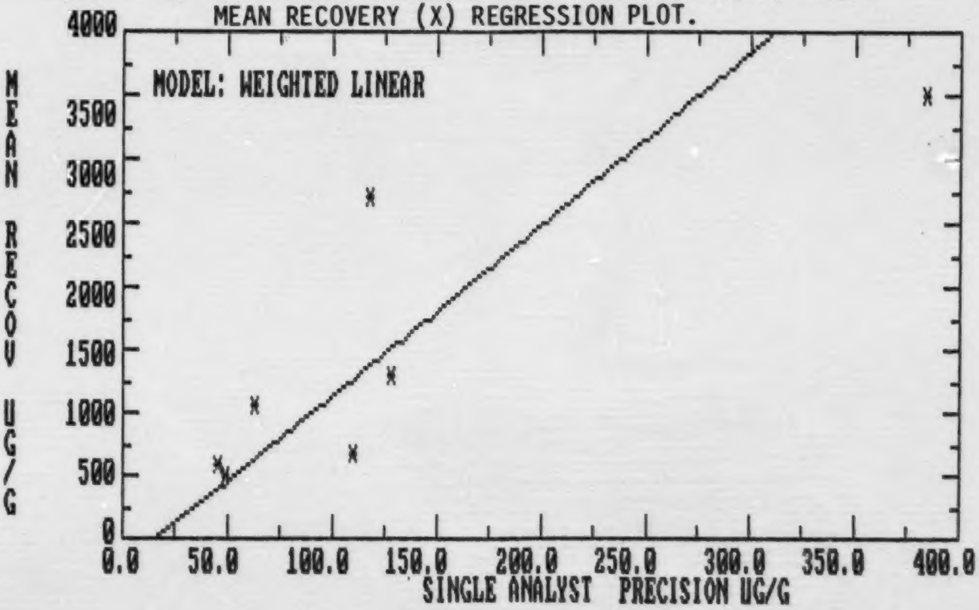
SR = 0.0618X - 6.25* Includes all seven solid wastes.

FIGURE 33. NICKEL: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



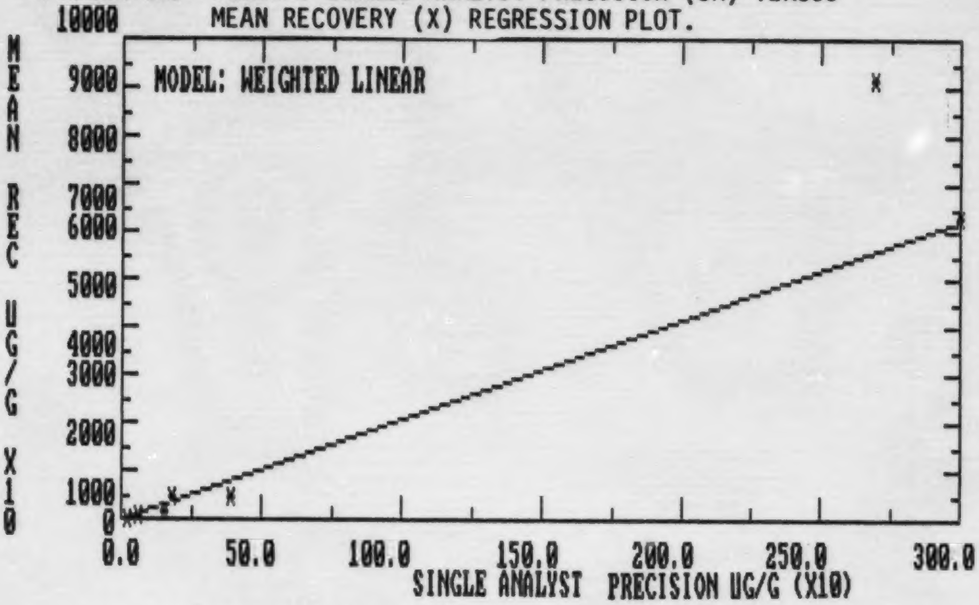
SR = 0.0438X + 5.64* Includes all seven solid wastes.

FIGURE 34. POTASSIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



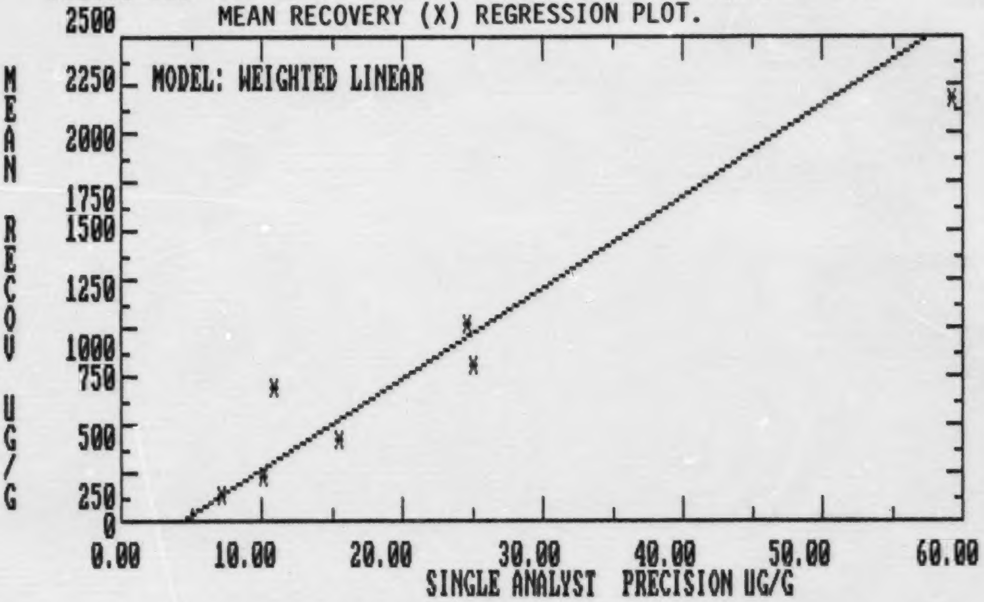
SR = 0.0739X + 16.14* Includes all seven solid wastes.

FIGURE 35. SODIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



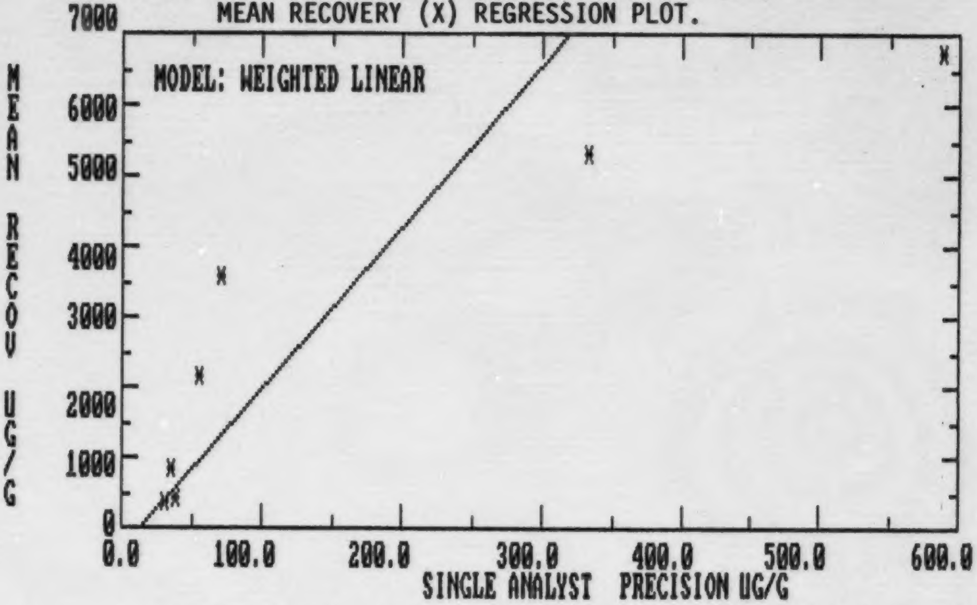
SR = 0.0481X + 8.39* Includes all seven solid wastes.

FIGURE 36. THALLIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



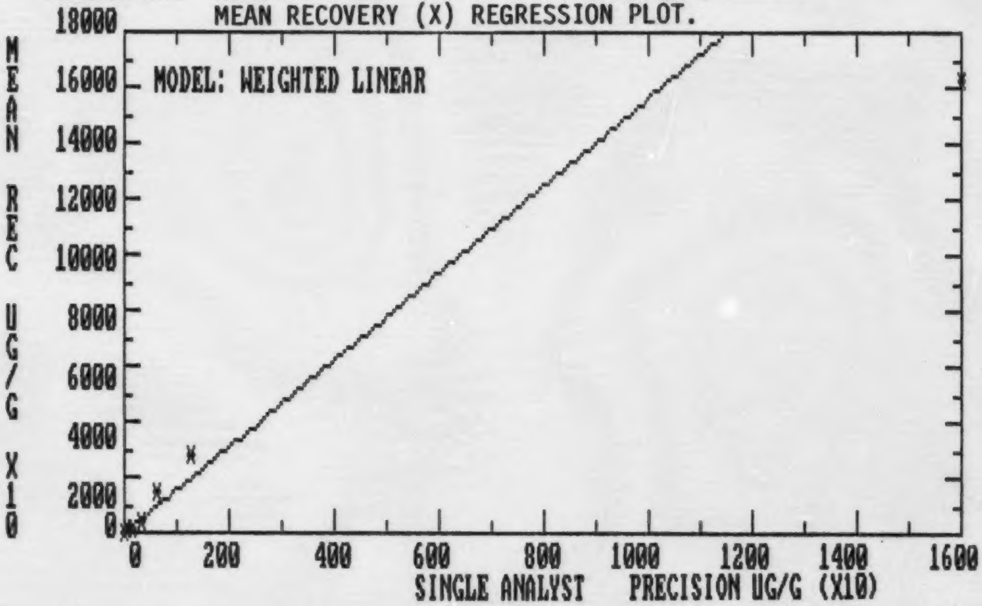
SR = 0.0213X + 4.33* Includes all seven solid wastes.

FIGURE 37. VANADIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



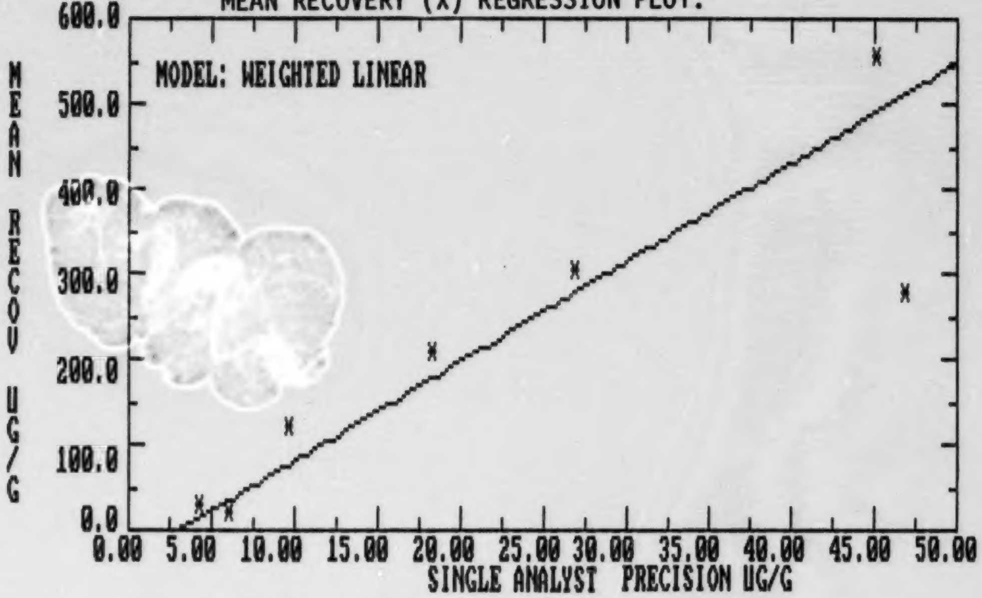
SR = 0.0437X + 13.6* Includes all seven solid wastes.

FIGURE 38. ZINC: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



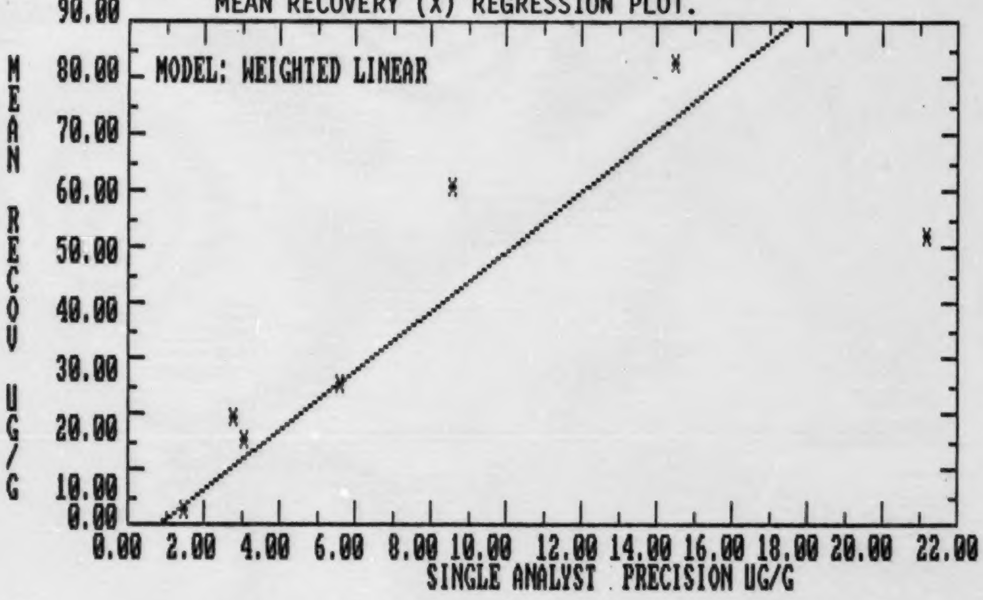
SR = 0.0639X + 1.33* Includes all seven solid wastes.

FIGURE 39. ARSENIC: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



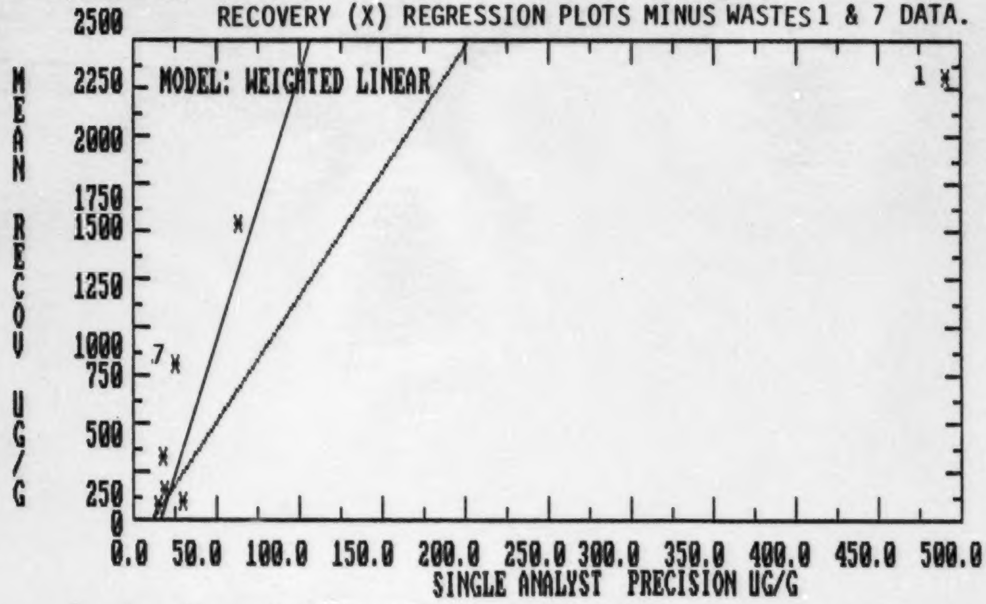
SR = 0.0857X + 2.96* Includes all seven solid wastes.

FIGURE 40. SELENIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



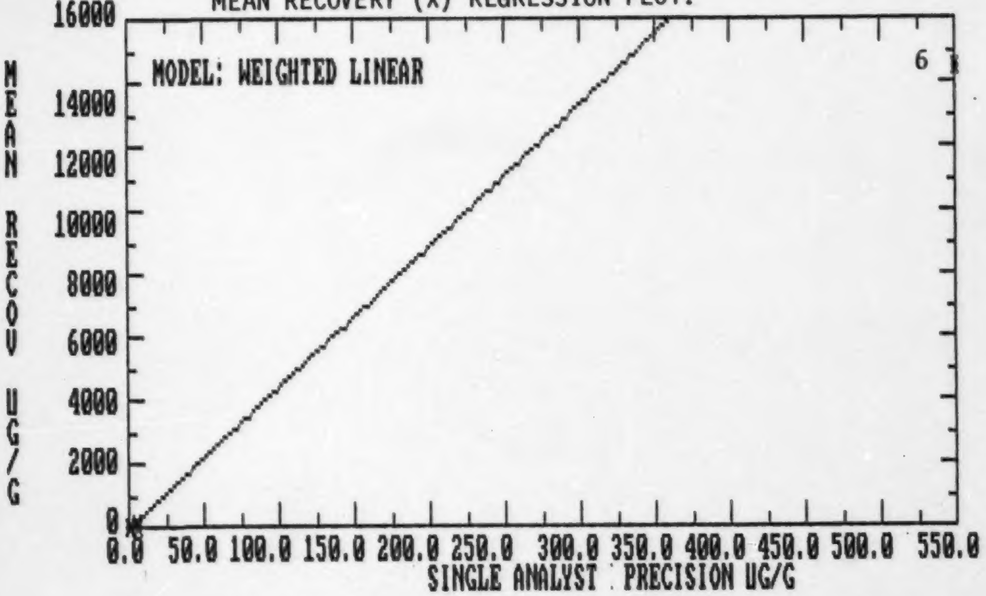
SR = 0.187X + 0.85* Includes all seven solid wastes.

FIGURE 41. ANTIMONY: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 1 & 7 DATA.



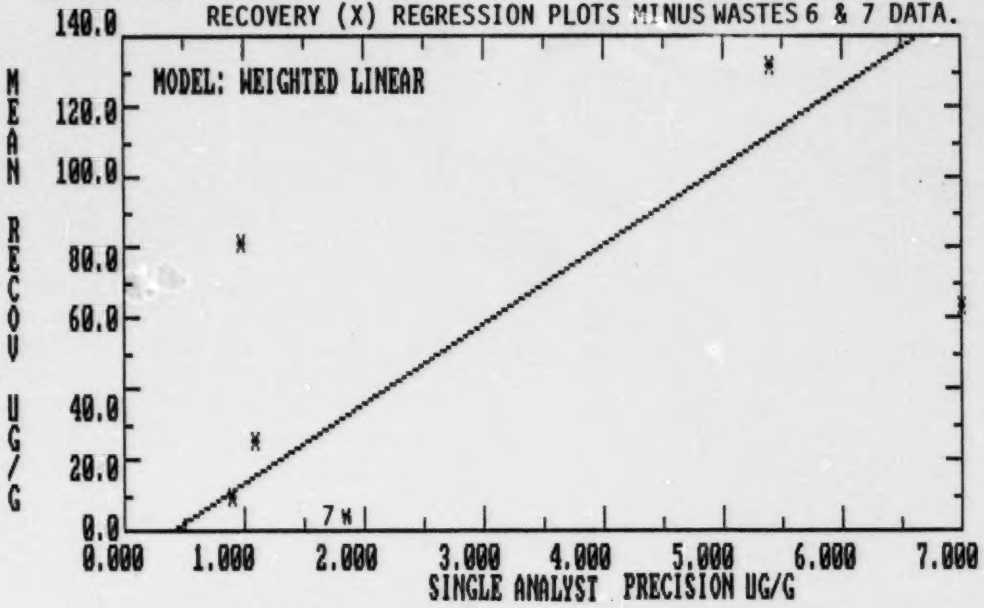
(---) SR = 0.0765X + 11.40 Includes all seven solid wastes.
(——) SR = 0.0310X + 16.35* Solid wastes 1,7 removed.

FIGURE 42. CADMIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



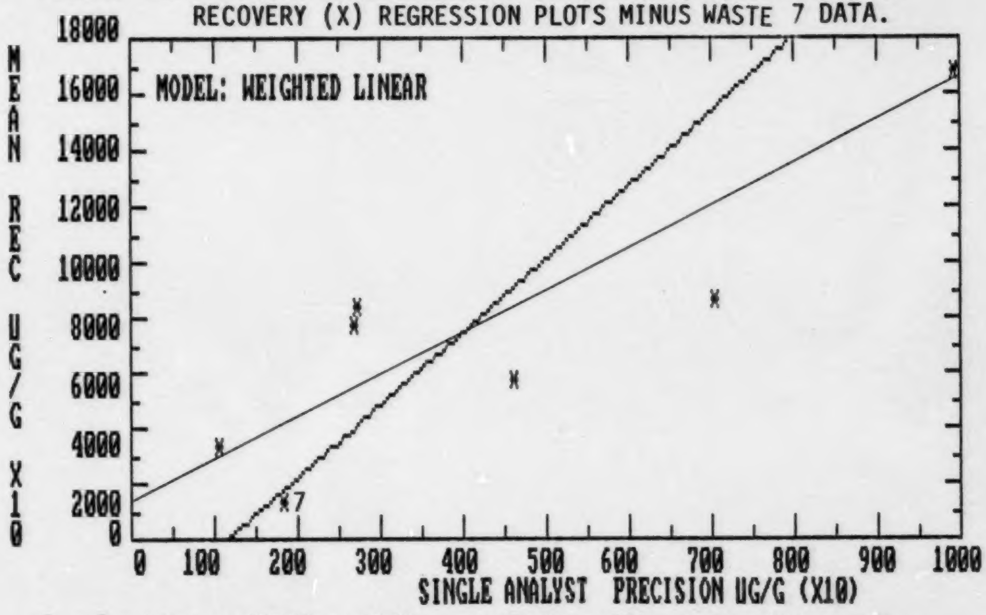
SR = 0.0225X + 1.40 Includes all seven solid wastes.

FIGURE 43. CADMIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 6 & 7 DATA.



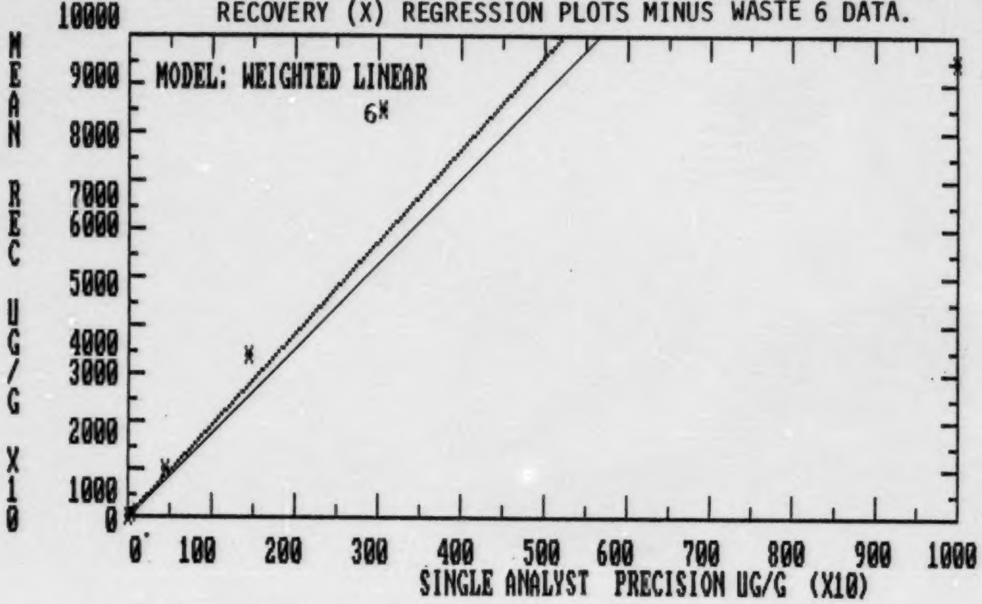
SR = 0.0446X + 0.41* Solid wastes 6,7 removed.

FIGURE 44. CALCIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 7 DATA.



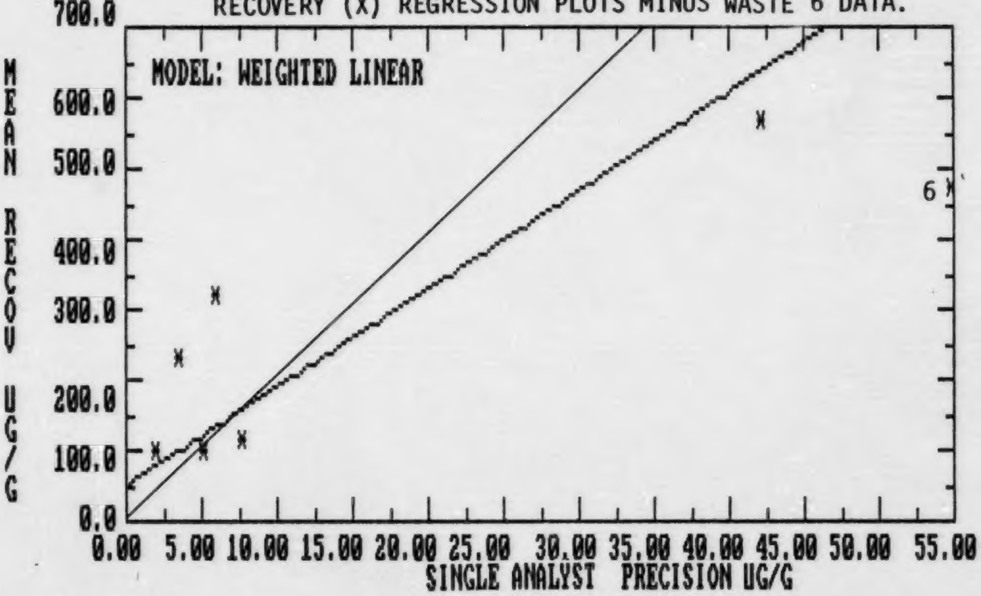
(---) SR = 0.0379X + 1156 Includes all seven solid wastes.
(——) SR = 0.0662X - 881.1* Solid Waste 7 removed.

FIGURE 45. CHROMIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 6 DATA.



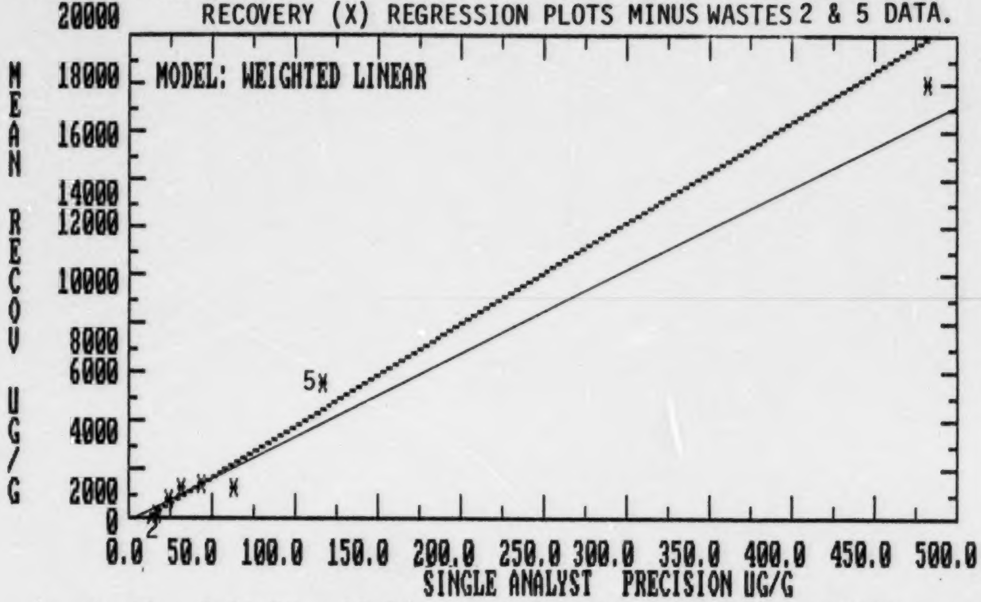
(---) SR = 0.0524X + 1.10 Includes all seven solid wastes.
(——) SR = 0.0564X + 0.66* Solid Waste 6 removed.

FIGURE 46. COBALT: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 6 DATA.



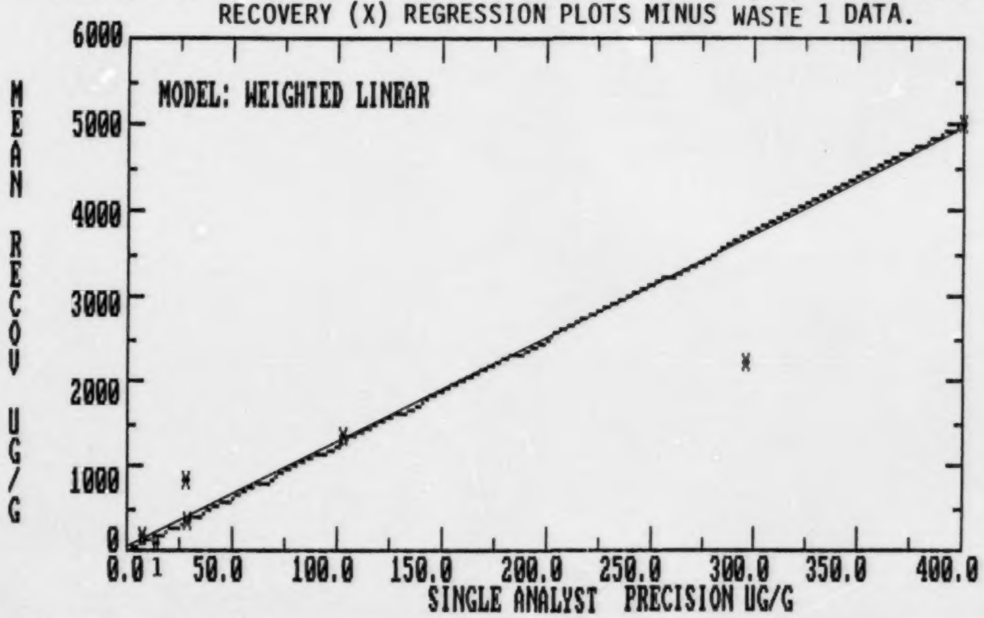
(---) SR = 0.0718X - 3.74 Includes all seven solid wastes.
(——) SR = 0.0428X - 0.40* Solid Waste 6 removed.

FIGURE 47. LEAD: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 2 & 5 DATA.



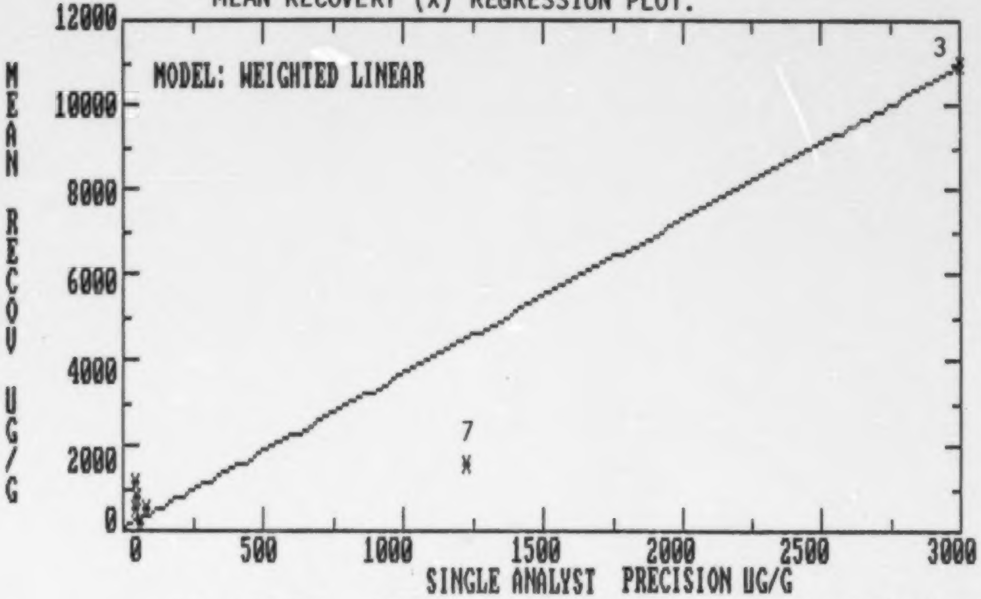
(---) SR = 0.0237X + 10.84 Includes all seven solid wastes.
(——) SR = 0.0292X + 4.33* Solid Wastes 2,5 removed.

FIGURE 48. MOLYBDENUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTE 1 DATA.



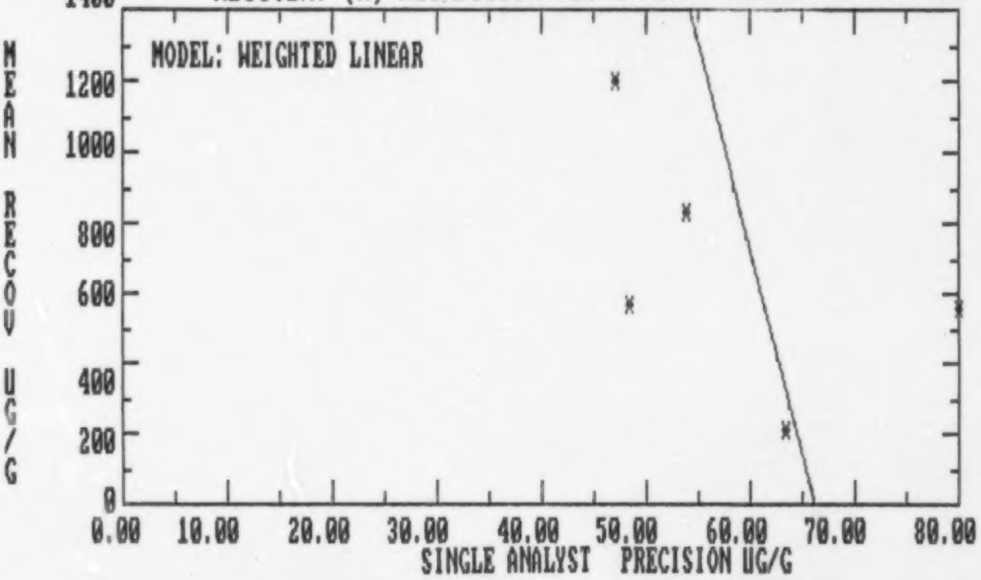
(---) SR = 0.0798X + 0.30 Includes all seven solid wastes.
(——) SR = 0.0936X - 10.75* Solid waste 1 removed.

FIGURE 49. BARIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOT.



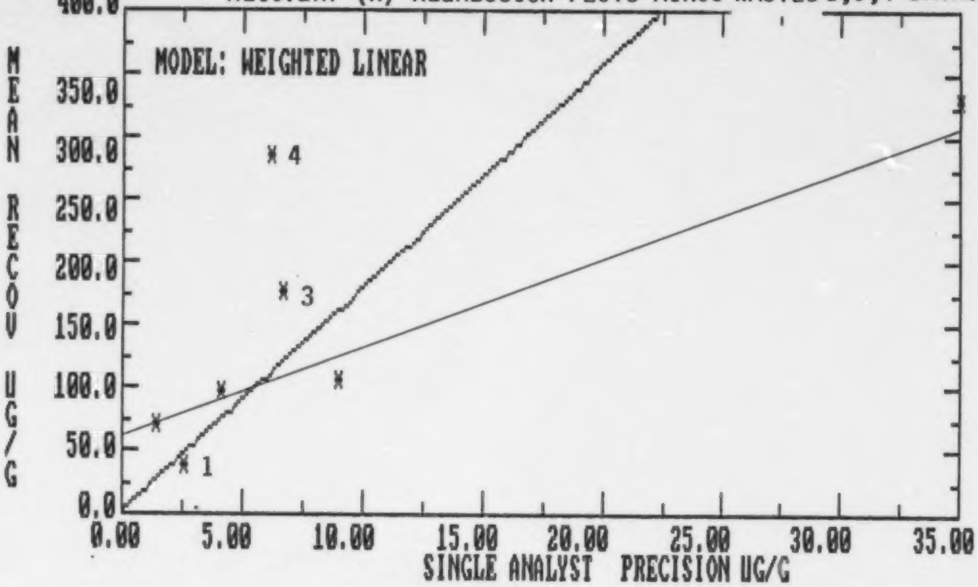
SR = 0.277X - 22.54 Includes all seven solid wastes.

FIGURE 50. BARIUM: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 3 & 7 DATA.



SR = -0.0086X + 66.25 Solid Wastes 3,7 removed.
No equation is favored. Data is too variable.

FIGURE 51. SILVER: SINGLE-ANALYST PRECISION (SR) VERSUS MEAN RECOVERY (X) REGRESSION PLOTS MINUS WASTES 1,3,4 DATA.



(---) SR = 0.0562X - 0.12 Includes all seven solid wastes.
(——) SR = 0.141X - 8.39 Solid wastes 1,3,4 removed.
No equation is favored-three solid waste data points had to be removed.

REFERENCES

1. Youden, W.J. and Steiner, E.M., Statistical Manual of the Association of Official Analytical Chemists, AOAC, Arlington, VA, p.88 (1975).

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APPENDIX A

INTERLABORATORY METHOD VALIDATION STUDY

3050

LABORATORY INSTRUCTIONS

Interlaboratory Method Validation Study

3050

PLEASE READ ALL INSTRUCTIONS BEFORE PROCEEDING

Your laboratory reporting code will be:

____ ____ ____

This number is to be used on all data report forms.

Unpacking the Cooler

It is recommended that the cooler be unpacked and the sample bottles segregated into groups according to the packing list found in Table A1. If you find any sample bottles missing or leaking, call immediately for replacements.

Mr. Kenneth Edgell
The Bionetics Corporation
16 Triangle Park Drive
Cincinnati, OH 45246
(513) 771-0448

TABLE 6

METHOD 3050 - PACKING LIST

1. One set of instructions and data report forms.
2. Two laboratory notebooks.
3. Sample sets.

		SOLIDS 1	SOLIDS 2	SOLIDS 3	SOLIDS 4	SOLIDS 5	SOLIDS 6	SOLIDS 7
Mix 1	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 2	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 3	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 4	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B

# Bottles

56

4. RFP OPTION V

			# Bottles
a)	DIGESTATE 1	mix 1, mix 2, mix 3, mix 4	4
b)	DIGESTATE 2	mix 1, mix 2, mix 3, mix 4	4
c)	DIGESTATE 3	mix 1, mix 2, mix 3, mix 4	4
d)	DIGESTATE 4	mix 1, mix 2, mix 3, mix 4	4
e)	DIGESTATE 5	mix 1, mix 2, mix 3, mix 4	4
f)	DIGESTATE 6	mix 1, mix 2, mix 3, mix 4	4
g)	DIGESTATE 7	mix 1, mix 2, mix 3, mix 4	4
			28

5. US EPA Quality Control Samples

			# Ampules
U.S. EPA ICAP-19	WP1083	conc. 1	6
U.S. EPA ICAP-7	WP1083	conc. 1	2

1. INTRODUCTION

The purpose of this Interlaboratory Method Validation Study (IMVS) is to provide precision and accuracy statements for 23 metals in sludges, soils and sediments for Resource Conservation and Recovery Act (RCRA) method 3050. The 23 metals of interest are aluminum (Al), barium (Ba), beryllium (Be), calcium (CA), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), potassium (K), magnesium (mg), manganese (Mn), molybdenum (Mo), sodium (Na), nickel (Ni), antimony (Sb), vanadium (V), zinc (Zn), silver (Ag), thallium (Tl), arsenic (As) and selenium (Se).

The enclosed samples are composed of seven (7) solids matrices each containing four (4) groups of analytes.

For each SOLIDS matrix

Mix 1	analyze for	Al, Ca, Fe, Mg, Mn, K, Na
Mix 2	analyze for	Be, Cd, Cr, Co, Cu, Pb, Mo, Ni, Sb, Tl, V, Zn
Mix 3	analyze for	As, Se
Mix 4	analyze for	Ag, Ba

Each sample bottle has been spiked with the necessary elements. No further spiking will be necessary.

The design of the IMVS for method 3050 is based on Youden's original non-replicate design for collaborative evaluation of precision and accuracy for analytical methods.[1] According to Youden's design, two similar yet different samples are analyzed in pairs. For the IMVS of 3050 one pair will be used for each of seven SOLIDS types, totalling 14 samples for each mixture. The analyst is directed to do a single analysis and report one value for each analyte.

OPTION V of the Request for Proposal (RFP) is also being exercised for this study. The instructions for this option will be covered in later sections of this booklet.

Each contractor has 60 calendar days, from receipt of the sample, to return all analytical data. The address to which all data, and analytical questions, is to be returned is covered in the Data Report Forms section.

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[1] Youden, W.J. Statistical Techniques for Collaborative Tests. Association of Official Analytical Chemists, Inc., Washington, D.C.,1969

Two laboratory notebooks are being provided to each contractor for the collection of raw data and analytical procedure descriptions. The yellow tear out pages are for the contractor to keep. The notebooks are then to be returned, with the data report forms, at the completion of the study.

2. ANALYTICAL PROCEDURES

Before proceeding, make sure you have all the necessary sample bottles as outlined in TABLE A2.

2.1 Sample Digestion

Percent moisture determinations are not required for this IMVS. All samples have been thoroughly dried and exactly weighed into each bottle.

Quantitatively transfer the entire contents of each bottle for digestion. All sample bottles labelled Mix 1 contain dried solids. Bottles labelled Mix 2, Mix 3 and Mix 4 contain solids plus a liquid layer. These samples are also to be quantitatively transferred.

A copy of SW-846 method 3050 is attached to this booklet. Digest all samples, quality control samples and procedural blanks according to sections 7.1 through 7.4 of method 3050. At this point, special precautions must be observed. Samples from bottles labelled Mix 1, Mix 2 and Mix 4 will be further digested according to section 7.5 using HCl. The bottles labelled Mix 3 will proceed using section 7.6 continuing with the HNO_3 digestion.

TABLE 7. SAMPLES FOR DIGESTION

		SOLIDS 1	SOLIDS 2	SOLIDS 3	SOLIDS 4	SOLIDS 5	SOLIDS 6	SOLIDS 7
Mix 1	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 2	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 3	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B
Mix 4	Bottle	A	A	A	A	A	A	A
	Bottle	B	B	B	B	B	B	B

Digest samples in groups as suggested below.

	Digestions
Mix 1 Bottle A SOLIDS 1 to SOLIDS 2	7
EPA Quality Control Sample ICAP-19	1
EPA Quality Control Sample ICAP-7	1
Procedural Blank	1

This scheme will result in 8 digestion groups. Maintain the integrity of each group so that the QC samples and blank adequately reflect the analytical procedure the samples are subjected to. Further explanations about the use of QC samples and the procedural blank will be covered in subsequent sections.

Record all digestion procedures in the laboratory notebook provided. This should include volumes and type of acid used, the duration of heating cycle and hot plate settings. It is important that method 3050 be followed exactly for this is a method validation study not a method development study. Do not attempt to maximize any step in the digestion procedure based on past experience. Failure to follow the prescribed method can lead to nonpayment and/or forfeiture of your contract.

After the samples have been digested, filter, and dilute the digestate to 100mL using ASTM type II water. The sample is now ready for analysis.

2.2 Procedural Blank

The procedural blank will be your own laboratory reagent water digested according to method 3050. There will be one procedural blank with each group of SOLIDS digested. Refer to section 2.1 for the make up of a group. Each blank will be digested in the same manner as the rest of the samples in the group.

The blanks will be used to isolate systematic errors which may be introduced into the results based on laboratory procedure.

2.3 Quality Control Samples

There are two different U.S. EPA Quality Control samples being provided for this study. One is U.S.EPA ICAP-19 WP1083 conc 1, the other is U.S.EPA ICAP-7 WP 1083 conc 1. Dilution instructions are as follows:

ICAP-19 Pipet exactly 20 mL into 100 mL volumetric flask. Bring to volume with ASTM type II water.

ICAP-7 Pipet exactly 5 mL into a 100 mL volumetric flask. Bring to volume with ASTM type II water.

The reference values for these diluted QC samples, and the ± 20% acceptance limits, are found in Table A3.

QC samples must be digested with each sample group using the same digestion procedure as the samples. Since the IMVS 3050 is comprised of four mixes, each containing different elements, the enclosed QC samples will have to be divided in the following manner.

Mix 1 digestion groups require ICAP-19 and ICAP-7
Mix 2 digestion groups require ICAP-19
Mix 3 digestion groups require ICAP-19
Mix 4 digestion groups require ICAP-7

Once the QC samples are diluted they are ready for digestion. Quantitatively transfer the 100 milliliters of each QC sample into digestion beakers and follow the same digestion procedure as the group it is with. These QC samples are being used as an independent check on the entire analytical procedure, from digestion to instrumental analysis. If an analyte value falls outside of its acceptance limits the entire group of samples will have to be redigested and analyzed at the participating laboratory's expense.

Additional sample bottles and QC ampuls can be obtained by calling Mr. Kenneth Edgell at the Bionetics Corporation (513) 771-0448. Be extremely cautious during all digestion procedures.

A quality control sample report form (blue form) has been included with this study for the reporting of QC results. This form can be found, with the other data report forms, in the back of this booklet. Use the QC report form titled "IMVS 3050".

3. INSTRUMENTAL ANALYSIS

Instrumental analyses will be performed using conditions and methods specified in the EPA 7000 series methods and your instrument manual.

There will be no requirements for a specific analytical order because the element concentrations vary between the SOLIDS. No individual matrix contains all low or all high concentrations. It is recommended that the Bottle A and Bottle B samples be analyzed together for each of the SOLIDS e.g. SOLIDS 1 Bottle A, Bottle B. SOLIDS 2 Bottle A, Bottle B, etc.

All raw data must be recorded in the laboratory notebooks provided. This should include absorbance values obtained for standards and samples, as well as, the concentration values obtained. Mistakes in reporting data cannot be corrected if one or the other piece of information is missing. If your instrument provides computer generated tables with this information it is not necessary to transcribe it into the notebooks. Bind up the computer reports and submit it with the data report forms.

TABLE 8. REFERENCE VALUES FOR QC SAMPLES

ICAP-19

Parameter	Reference Value (mg/L)	Acceptance Limits (mg/L) 80%	Acceptance Limits (mg/L) 120%
Sb	1.98	1.58	2.38
Be	1.92	1.54	2.30
Cd	1.88	1.50	2.26
Ca	2.00	1.60	2.40
Cr	2.06	1.65	2.47
Cu	2.06	1.65	2.47
Co	2.00	1.60	2.40
Fe	2.04	1.63	2.45
Pb	2.02	1.62	2.42
Mg	2.00	1.60	2.40
Mn	2.04	1.63	2.45
Mo	2.12	1.70	2.54
Ni	2.04	1.63	2.45
Tl	2.02	1.62	2.42
V	2.02	1.62	2.42
Zn	2.02	1.62	2.42

ICAP-7

Parameter	Reference Value (mg/L)	Acceptance Limits (mg/L) 80%	Acceptance Limits (mg/L) 120%
Ag	4.95	3.96	5.94
Al	4.85	3.88	5.82
Ba	4.85	3.88	5.82
K	50.0	40.0	60.0
Na	4.30	3.44	5.16

WP 1083

4. Data Report Forms

There are three sets of data report forms contained in this instruction booklet. The main set (white pages) are the forms to use for the study 3050 results. A second set (yellow pages) contains report forms for the OPTION V results and the final set (blue pages) contains the quality control sample report form and the instrument identification form.

You will notice that for this study the report forms contain two elements per page. All data entry blocks are clearly titled. Make sure your laboratory code is placed one each page in blocks 77-79. At a minimum your laboratory name and the analyst's name should be included on each page.

All data is to be reported as total micrograms analyzed. No moisture determinations are required and no calculations are necessary for the reporting of concentrations versus the amount of solids present.

An analytical value MUST be reported. Contractors will not be paid for data reported as zero or less than MDL. There is sufficient analyte present in each of the samples to make analytical results obtainable by all contractors. This requirement also applies to the OPTION V digestate samples.

Numbers are to be right justified in the appropriate boxes. A decimal point will have its own box. No other symbols will be accepted in the boxes. If the concentrations exceed 1000, do not use a comma.

AMPUL 1

			1	2	.	2

1 - 7

AMPUL 2

		8	2	7	0	0

10 - 16

Only three significant figures should be reported according to the following format.

XXX00 XXX.X
XXX0 0.XX
XXX 0.0XX

At the completion of this study, return the laboratory notebooks and data report forms to:

Mr. Kenneth Edgell
The Bionetics Corporation
16 Triangle Park Drive
Cincinnati, OH 45246
(513) 771-0448

5. QUALITY CONTROL/QUALITY ASSURANCE REQUIREMENTS

Each participating laboratory will be required to implement quality control/quality assurance protocols for this study. These protocols must include, but are not limited to, the following procedures:

1. Preparation of calibration curves for each element. Each curve will consist of a blank and at least four calibration standards.

2. Analysis of check standards at the beginning and end of each working day and at a frequency of 10% during analytical runs. If analysis of a check standard is not within ±5% of the expected value, analysis must be terminated, the instrument must be recalibrated, and all samples run since last successful check standard must be reanalyzed.

3. Preparation and analysis of one procedural blank for every digestion group.

4. Analysis of an outside reference standard having known values. This sample will be provided by EPA and will be analyzed with each analytical group as covered in section 2.3 of this packet.

5. Maintenance of accurate written records that trace all samples from receipt through preparation, analysis, and final data submittal. Each contractor will be required to maintain all pertinent raw and final data until informed otherwise by EPA.

6. Maintenance of logs documenting instrument repairs or modifications performed during the time frame of this study.

METHOD 3050

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also Paragraph 2.1):

FLAA		GFAA
Aluminum	Magnesium	Arsenic
Barium	Manganese	Beryllium
Beryllium	Molybdenum	Cadmium
Cadmium	Nickel	Chromium
Calcium	Potassium	Cobalt
Chromium	Sodium	Lead
Cobalt	Thallium	Molybdenum
Copper	Vanadium	Selenium
Iron	Zinc	Thallium
Lead		Vanadium

2.0 SUMMARY OF METHOD

2.1 A representative 1- to 2-g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, and (2) the flame AA or ICP analysis of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Mo, Pb, Ni, K, Na, Tl, V, and Zn. Dilute nitric acid is employed as the final dilution acid for the furnace AA analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V. A separate sample shall be dried for a total solids determination.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method 3050 is applicable to a given waste.

4.0 APPARATUS AND MATERIALS

4.1 Conical Phillips beakers: 250-mL.
4.2 Watch glasses.
4.3 Drying ovens: That can be maintained at 30°C.
4.4 Thermometer: That covers range of 0 to 200°C.
4.5 Whatman No. 41 filter paper (or equivalent)
4.6 Centrifuge and centrifuge tubes.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrate nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.

5.3 Concentrated hydrochloric acid, reagent grade (HCl): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.

5.4 Hydrogen peroxide (30%) (H_2O_2): Oxidant should be analyzed to determine level of impurities.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable. See Chapter Three, Section 3.1.3, for further information.

6.3 Nonaqueous samples shall be refrigerated upon receipt and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.01 g and transfer to a conical beaker a 1.00- to 2.00-g portion of sample.

7.2 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 to 15 min. without boiling.

7.2 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 to 15 min. without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min. Repeat this last step to ensure complete oxidation.

Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

7.3 After Step 7.2 has been completed and the sample has cooled, add 2 mL of Type II water add 3 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

7.4 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

7.5 If the sample is being prepared for (a) the ICP analysis of As and Se, or (b) the flame AA or ICP analysis of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Na, Tl, V, and Zn, then add 5 mL of concentrated HCl and 10 mL of Type II water. Particulates in the digestate that may clog the nebulizer should be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.5.1 Filtration: Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with Type II water.

7.5.2 Centrifugation: Centrifugation at 2,000-3,000 rpm for 10 min. is usually sufficient to clear the supernatant.

7.5.3 The diluted sample has an approximate acid concentration of 5.0% (v/v) HCl and 5.0% (v/v) HNO_3. The sample is now ready for analysis.

7.6 If the sample is being prepared for the furnace analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V, cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL. After cooling, dilute to 100 mL with Type II water. Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.6.1 Filtration: Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 mL with Type II water.

7.6.2 Centrifugation: Centrifugation at 2,000-3,000 for 10 min. is usually sufficient to clear the supernatant.

7.6.3 The diluted digestate solution contains approximately 5% (v/v) HNO_3. For analysis, withdraw aliquots of appropriate volume and add any required reagent or matrix modifier. The sample is now ready for analysis.

7.7 Calculations:

7.7.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

7.7.2 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

8.0 QUALITY CONTROL

8.1 For each group of samples processed, preparation blanks (Type II water and reagents) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

8.2 Duplicate samples should be processed on a routine basis. Duplicate samples will be used to determine precision. The sample load will dictate the frequency, but 20% is recommended.

8.3 Spiked samples or standard reference materials must be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

8.4 The concentration of all calibration standards should be verified against a quality control check sample obtained from an outside source.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

10.1 None required.

APPENDIX B

RAW DATA

TABLE 9. RAW DATA FOR ALUMINUM,AL

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		27400.0	33400.0	42300.0	59200.0	25200.0	35300.0	16900.0	23700.0
1		4910.0	6460.0	5500.0	6590.0	12300.0	18600.0	4890.0	6830.0
2		3990.0	5180.0	5040.0	6740.0	12900.0	17900.0	4890.0	6790.0
3		4620.0	5670.0	6160.0	8770.0	15400.0	19900.0	6970.0	11900.0
4		4130.0	5850.0	5690.0	7020.0	11800.0	17400.0	4410.0	6100.0
5		5510.0	7340.0	7830.0	11000.0	16200.0	22200.0	7920.0	11200.0
6		6770.0	9470.0	9750.0	12400.0	17200.0	23800.0	10200.0	14200.0
7		4350.0	6300.0	5650.0	8150.0	12300.0	15200.0	6090.0	8900.0
8		3050.0	3910.0	5100.0	7340.0	11600.0	14500.0	4590.0	6310.0

RAW DATA FOR ALUMINUM,AL

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		41700.00	58400.00	1430.00	2000.00	5100.00	7140.00
1		8520.00	12800.00	930.00	1380.00	1490.00	1940.00
2		6850.00	9520.00	682.00	798.00	1330.00	1650.00
3		7810.00	14100.00	660.00	850.00	1420.00	1870.00
4		8600.00	11300.00	645.00	614.00	1110.00	1990.00
5		10300.00	15300.00	744.00	799.00	1370.00	1760.00
6		13400.00*	18400.00	1000.00	1320.00	1920.00	2630.00
7		9120.00	13200.00	685.00	912.00	1200.00	1720.00
8		8730.00	12500.00	454.00	570.00	918.00	1260.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

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TABLE 10. RAW DATA FOR ANTIMONY,SB

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		3937.0	4912.0	221.0	274.0	423.0	528.0	1935.0	2415.0
LAB/AN	LAB REJECTED								
1		3170.0	2180.0	107.0	76.0	160.0	159.0	1590.0	1900.0
2		2820.0	3620.0	114.0	132.0	114.0	151.0	1630.0	1830.0
3		2660.0	3340.0	111.0	127.0	139.0	149.0	1420.0	1600.0
4		990.0	357.0	322.0*	322.0*	335.0*	256.0	1070.0	1390.0
5		3260.0	2980.0	121.0	67.6	102.0	151.0	1580.0	1760.0
6		1580.0	1220.0	98.7	139.0	164.0	155.0	1490.0	1800.0
7		3120.0	4040.0	138.0	170.0	213.0	223.0	1400.0	1750.0
8		861.0	389.0	123.0	59.0	119.0	179.0	1110.0	1550.0

RAW DATA FOR ANTIMONY,SB

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		603.00	753.00	140.00	196.00	920.00	1145.00
LAB/AN	LAB REJECTED						
1		321.00	303.00	88.00	101.00	769.00	913.00
2		319.00	343.00	64.00	88.80	778.00	995.00
3		359.00	355.00	54.00	56.00	714.00	859.00
4		442.00	482.00	343.00*	482.00*	910.00*	1180.00*
5		316.00	373.00	38.70	64.70	697.00	899.00
6		245.00	277.00	82.50	107.00	739.00	885.00
7		362.00	383.00	53.20	117.00	692.00	810.00
8		300.00	290.00	59.00	109.00	716.00	868.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 11. RAW DATA FOR BARIUM,BA

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		1080.0	1280.0	1390.0	1616.0	209904.0	212380.0	209904.0	292380.0
1		762.0	969.0	990.0	1360.0	5510.0	10500.0	49.0	69.0
2		768.0	970.0	1060.0	1380.0	7870.0	10200.0	130.0	86.9
3		703.0	915.0	955.0	1200.0	16900.0	12600.0	643.0	473.0*
4		817.0	925.0	1060.0	1510.0	14400.0	9880.0	309.0	237.0
5		752.0	1030.0	1150.0	1460.0	11100.0	12800.0	392.0	169.0
6		1250.0*	1410.0*	2720.0*	3830.0*	14800.0	14900.0	816.0	1090.0*
7		726.0	852.0	978.0	1360.0	9780.0	9960.0	124.0	69.1
8		622.0	945.0	1030.0	1330.0	2410.0	12200.0	84.0	107.0

RAW DATA FOR BARIUM,BA

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		640.00	896.00	480.00	672.00	5252.00	6490.00
1		483.00	757.00	558.00	660.00	732.00	1750.00
2		496.00	697.00	490.00	697.00	5030.00	370.00
3		244.00*	302.00	397.00	325.00	2940.00	462.00
4		1250.00*	1860.00*	645.00	818.00	2130.00	254.00
5		561.00	845.00	554.00	834.00	3670.00	2760.00
6		2750.00*	3860.00*	1810.00*	2510.00*	2890.00	1230.00
7		466.00	662.00	489.00	665.00	583.00	327.00
8		475.00	590.00	376.00	403.00	291.00	176.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 12. RAW DATA FOR BERYLLIUM,BE

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		19.2	24.0	38.4	48.0	198.4	248.0	144.0	180.0
1		18.0	23.0	34.0	43.0	165.0	224.0	135.0	163.0
2		16.8	21.2	36.4	44.5	187.0	226.0	133.0	162.0
3		9.0*	24.0	28.0	48.0	194.0	243.0	176.0	177.0
4		18.7	21.7	33.0	41.6	180.0	224.0	134.0	154.0
5		19.2	23.8	40.0	49.0	191.0	244.0	151.0	187.0
6		19.3	23.6	39.6	45.2	214.0	238.0	156.0	198.0
7		19.4	22.0	36.6	43.1	184.0	220.0	137.0	170.0
8		18.3	22.4	36.3	44.3	139.0	229.0	141.0	169.0

RAW DATA FOR BERYLLIUM,BE

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		4.80	6.00	96.00	120.00	80.00	100.00
1		4.60	6.50	88.00	106.00	72.00	89.00
2		4.43	5.29	87.20	103.00	80.00	99.60
3		5.08	7.00	89.00	119.00	74.00	95.00
4		6.40	7.11	83.80	105.00	76.30	94.90
5		5.77	6.99	95.40	123.00	82.60	106.00
6		6.23	7.30	104.00	125.00	91.70	108.00
7		6.72	6.34	84.90	102.00	75.90	85.70
8		4.70	5.90	85.20	106.00	76.70	99.30

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 13. RAW DATA FOR CADMIUM,CD

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		10.2	14.3	59.6	74.5	20.0	28.0	110.0	154.0
LAB/AN	LAB REJECTED								
1		7.4	12.0	49.0*	59.0	14.0*	30.0	105.0	146.0
2		7.9	11.1	61.0	75.3	21.1	29.4	115.0	162.0
3		7.7	9.9	59.9	74.0	21.1	29.0	158.0*	113.0*
4		14.6	15.6	62.5	75.0	20.3	31.2	109.0	146.0
5		11.8	15.8	63.1	72.2	24.2	33.1	117.0	158.0
6		7.3	10.8	58.9	45.8	20.4	17.8*	95.1	152.0
7		8.9	10.7	60.5	68.0	23.2	32.7	114.0	169.0
8		6.4	8.1	58.0	74.0	22.1	28.1	108.0	150.0

RAW DATA FOR CADMIUM,CD

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		74.40	93.00	12500.00	17500.00	5.96	7.45
LAB/AN	LAB REJECTED						
1		68.00	85.00	11600.00	15700.00	6.00	1.70
2		74.50	92.00	12600.00	17800.00	2.49	3.35
3		70.40	89.60	10900.00	17100.00	4.30	5.30
4		75.00	93.80	11300.00	18000.00	10.40	10.40
5		77.20	97.80	13000.00	18500.00	7.98	10.70
6		71.10	88.20	11800.00	16800.00	2.54	2.54
7		78.90	95.60	11500.00	17100.00	5.36	8.93
8		68.30	87.50	11300.00	16700.00	< 5.00*	< 5.00*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 14. RAW DATA FOR CALCIUM,CA

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		28900.0	40500.0	75000.0	105000.0	68000.0	95200.0	53000.0	74200.0
1		27700.0	37100.0	69500.0	93500.0	61500.0	89200.0	49000.0	66400.0
2		28600.0	40300.0	72800.0	111000.0	70000.0	94600.0	44400.0	49100.0
3		28300.0	40700.0	77000.0	104000.0	68000.0	95000.0	52000.0	75000.0
4		20900.0*	25400.0*	61300.0	98900.0	58300.0	79100.0	42600.0	56600.0
5		30300.0	42900.0	69300.0	118000.0	74400.0	107000.0	56900.0	79200.0
6		27400.0	37700.0	73700.0	98100.0	67600.0	92500.0	49400.0	69300.0
7		26500.0	38200.0	65500.0	88500.0	60600.0	88500.0	44200.0	69600.0
8		27500.0	36500.0	70400.0	97000.0	64300.0	85300.0	49300.0	65500.0

RAW DATA FOR CALCIUM,CA

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		140000.00	196000.00	69200.00	96900.00	14900.00	20900.00
1		135000.00	194000.00	69300.00	96600.00	12900.00	18000.00
2		145000.00	125000.00	66200.00	100000.00	5670.00	5820.00
3		147800.00	195000.00	75000.00	104000.00	14000.00	13700.00
4		149000.00	115000.00	34800.00*	34800.00*	7840.00	10200.00
5		154000.00	77900.00*	79600.00	113000.00	15300.00	20400.00
6		131000.00	185000.00	71900.00	96900.00	13000.00	18700.00
7		133000.00	177000.00	62200.00	90100.00	13500.00	20200.00
8		135000.00	174000.00	69600.00	93300.00	13300.00	17200.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN
CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 15 . RAW DATA FOR CHROMIUM,CR

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		28700.0	40100.0	180.0	252.0	92.0	129.0	8100.0	11332.0
LAB/AN	LAB REJECTED								
1		21800.0	38000.0	90.0	132.0	51.0	62.0	8500.0	11800.0
2		29100.0	42200.0	142.0	194.0	79.1	110.0	8760.0	12200.0
3		29400.0	39900.0	132.0	134.0	71.0	101.0	9060.0	12600.0
4		32800.0	45800.0	162.0	199.0	93.3	99.1	8740.0	10900.0
5		28700.0	39800.0	146.0	198.0	63.4	95.1	8290.0	11700.0
6		29900.0	41600.0	137.0	200.0	70.1	104.0	8540.0	11800.0
7		27020.0	37900.0	95.0	133.0	55.0	72.5	8250.0	12800.0
8		27600.0	37300.0	130.0	134.0	67.0	90.0	7930.0	11400.0

RAW DATA FOR CHROMIUM,CR

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		1004.00	1405.00	66000.00	92400.00	80000.00	112000.00
LAB/AN	LAB REJECTED						
1		920.00	1180.00	72000.00	92000.00	78000.00	109000.00
2		1160.00	1420.00	72700.00	103000.00	85000.00	119000.00
3		1140.00	1450.00	72200.00	98800.00	85200.00	84800.00
4		1040.00	1260.00	70500.00*	99400.00	91500.00	138000.00
5		1170.00	1460.00	72700.00	102000.00	83700.00	117000.00
6		1220.00	1540.00	72100.00	95000.00	8890.00*	116000.00
7		1050.00	1200.00	67400.00*	100000.00	60000.00	110000.00
8		1120.00	1410.00	61200.00*	91200.00	73300.00	104000.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 16. RAW DATA FOR COBALT,CO

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		238.2	358.2	112.0	136.0	513.0	637.0	204.0	254.4
LAB/AN	LAB REJECTED								
1		263.0	331.0	80.0	93.0	269.0*	583.0	191.0	233.0
2		293.0	355.0	105.0	129.0	514.0	640.0	210.0	255.0
3		278.0	340.0	100.0	121.0	479.0	587.0	201.0	247.0
4		321.0	409.0	132.0	178.0	455.0	686.0	248.0	289.0
5		309.0	379.0	119.0	133.0	536.0	668.0	228.0	278.0
6		321.0	387.0	110.0	135.0	549.0	373.0*	219.0	268.0
7		290.0	353.0	106.0	120.0	537.0	593.0	200.0	238.0
8		284.0	350.0	106.0	130.0	525.0	611.0	192.0	228.0

RAW DATA FOR COBALT,CO

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		90.80	110.80	417.00	517.00	87.00	121.00
LAB/AN	LAB REJECTED						
1		81.00	103.00	383.00	465.00	78.00	118.00
2		88.80	112.00	406.00	496.00	95.40	120.00
3		83.00	103.00	398.00	477.00	93.00	124.00
4		128.00	183.00*	487.00	616.00	155.00*	225.00*
5		107.00	136.00*	443.00	752.00	115.00	155.00*
6		81.70	110.00	465.00	570.00	78.20	120.00
7		94.40	117.00	370.00	445.00	92.60	117.00
8		87.70	110.00	360.00	477.00	82.70	114.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 17. RAW DATA FOR COPPER,CU

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		120.0	168.0	5004.0	6205.0	450.0	630.0	810.0	1130.0
LAB/AN	LAB REJECTED								
1		135.0	199.0	4310.0	5330.0	343.0	553.0	800.0	1090.0
2		147.0	217.0	5130.0	6410.0	384.0	591.0	869.0	1210.0
3		146.0	195.0	5670.0	6450.0	373.0	565.0	810.0	1130.0
4		140.0	220.0	4740.0	6030.0	328.0	562.0	722.0	1030.0
5		148.0	214.0	4870.0	5750.0	376.0	570.0	832.0	1150.0
6		149.0	245.0	5850.0	6850.0	384.0	552.0	884.0	1310.0
7		138.0	225.0	4670.0	5600.0	357.0	560.0	786.0	1000.0
8		143.0	232.0	5210.0	6490.0	377.0	593.0	850.0	1180.0

RAW DATA FOR COPPER,CU

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		105.00	147.00	18600.00	25900.00	2700.00	3780.00
LAB/AN	LAB REJECTED						
1		63.00	79.00	18200.00	23900.00	1650.00	1620.00
2		68.10	87.20	19600.00	28000.00	1590.00	2200.00
3		61.00	71.00	17900.00	25600.00	1410.00	2040.00
4		69.40	69.40	19300.00	24700.00	1520.00	2290.00
5		64.20	88.30	18500.00	26300.00	1590.00	2230.00
6		684.00*	913.00*	20100.00	26600.00	2180.00*	3000.00
7		-69.00	84.50	17600.00	25200.00	1450.00	2000.00
8		67.90	84.40	17700.00	26400.00	1520.00	2070.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 18 . RAW DATA FOR IRON,FE

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		107000.0	150000.0	37000.0	51900.0	19100.0	26700.0	38100.0	53300.0
1		80400.0	118000.0	17300.0	22600.0	15900.0	23000.0	30600.0	41200.0
2		89300.0	122000.0	22000.0	28000.0	18200.0	25000.0	33600.0	44600.0
3		94900.0	134000.0	23600.0	29200.0	17800.0	25200.0	36200.0	52000.0
4		89100.0	131000.0	19000.0	23100.0	16400.0	21400.0	32700.0	45200.0
5		89300.0	122000.0	21500.0	28900.0	17600.0	24200.0	34400.0	48400.0
6		96900.0	125000.0	21200.0	26600.0	17200.0	22500.0	37900.0	47500.0
7		92200.0	123000.0	19600.0	24200.0	13800.0	21100.0	32200.0	46100.0
8		81600.0	112000.0	19300.0	26500.0	18000.0	25100.0	31700.0	47700.0

RAW DATA FOR IRON,FE

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		23900.00	33500.00	59100.00	82700.00	85000.00	X19000.00
1		20300.00	28600.00	59400.00	81400.00	78900.00	X11000.00
2		18400.00	30700.00	63200.00	87300.00	83400.00	X14000.00
3		22400.00	31200.00	62000.00	87400.00	81600.00	X16000.00
4		18500.00	23300.00	57800.00	57200.00*	75200.00	98100.00
5		21600.00	48600.00*	59300.00	30500.00	79600.00	X14000.00
6		20500.00	27600.00	61300.00	77500.00	72500.00	X13000.00
7		20300.00	29500.00	53700.00	76400.00	72900.00	X06000.00
8		20800.00	30400.00	60900.00	90700.00	82800.00	X15000.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 19. RAW DATA FOR LEAD,PB

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		730.0	1020.0	230.0	320.0	15200.0	21300.0	1180.0	1650.0
1		709.0	915.0	206.0	284.0	14400.0	20100.0	1290.0	1680.0
2		723.0	984.0	205.0	300.0	15100.0	20600.0	1290.0	1660.0
3		706.0	975.0	220.0	294.0	15700.0	20900.0	1190.0	1490.0
4		751.0	1020.0	290.0	354.0	15800.0	20500.0	1270.0	1590.0
5		747.0	1050.0	276.0	434.0*	16600.0	22800.0	1270.0	1600.0
6		686.0	391.0	215.0	331.0	15300.0	22600.0	1150.0	1640.0
7		700.0	972.0	196.0	330.0	13800.0	18900.0	1220.0	1540.0
8		697.0	958.0	231.0	332.0	14800.0	19900.0	1130.0	1560.0

RAW DATA FOR LEAD,PB

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		5033.00	6292.00	1020.00	1430.00	930.00	1300.00
1		5010.00	6250.00	1060.00	1440.00	1040.00	1390.00
2		4990.00	6200.00	1090.00	1520.00	1040.00	1470.00
3		5020.00	6320.00	1070.00	1470.00	1030.00	1440.00
4		5200.00	5960.00*	1120.00	1590.00	1100.00	1610.00
5		5860.00*	7230.00*	1150.00	1620.00	1130.00	1560.00
6		5380.00	6270.00	1040.00	1530.00	988.00	1450.00
7		5080.00	6190.00	1070.00	1480.00	1110.00	1330.00
8		4970.00	6320.00	1010.00	1510.00	1010.00	1480.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 20 RAW DATA FOR MAGNESIUM,MG

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		8400.0	11800.0	32000.0	44300.0	2300.0	3220.0	2000.0	2800.0
LAB/AN	LAB REJECTED								
1		6500.0	9000.0	25300.0	34800.0	2300.0	3400.0	1900.0	2700.0
2		5970.0	6230.0	25600.0	35700.0	2180.0	3050.0	1840.0	2510.0
3		6900.0	10200.0	27000.0	35500.0	2660.0	3710.0	2420.0	3530.0
4		7600.0	10400.0	24800.0	33400.0	2540.0	3500.0	2040.0	2750.0
5		6550.0	9150.0	26300.0	36300.0	2550.0	3690.0	2320.0	3250.0
6		7580.0	7370.0	24300.0	31200.0	2700.0	3200.0	2000.0	2820.0
7		3520.0*	9950.0	25700.0	35400.0	2080.0	3030.0	2080.0	2910.0
8		6480.0	8640.0	24500.0	35200.0	2480.0	3530.0	1800.0	2830.0

RAW DATA FOR MAGNESIUM,MG

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		43000.00	60200.00	19200.00	26900.00	1900.00	2660.00
LAB/AN	LAB REJECTED						
1		38100.00	53700.00	17300.00	24500.00	2300.00	3300.00
2		37800.00	53300.00	17100.00	24000.00	2110.00	2540.00
3		39200.00	53300.00	18200.00	26500.00	2590.00	3570.00
4		39000.00	52100.00	17000.00	17100.00*	2300.00	3080.00
5		37400.00	3160.00*	17500.00	24500.00	2470.00	3300.00
6		33700.00	43600.00	15500.00	22300.00	1980.00	2800.00
7		40100.00	50400.00	17800.00	25400.00	2430.00	3260.00
8		35400.00	48400.00	16600.00	24200.00	2360.00	3270.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 21 . RAW DATA FOR MANGANESE,MN

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		750.0	1050.0	550.0	770.0	810.0	1130.0	330.0	460.0
LAB/AN	LAB REJECTED								
1		782.0	1150.0	174.0*	227.0*	164.0*	226.0*	307.0	415.0
2		732.0	1030.0	421.0	571.0	831.0	1160.0	298.0	423.0
3		685.0	926.0	422.0	533.0	756.0	1090.0	284.0	389.0
4		736.0	1040.0	407.0	533.0	831.0	1120.0	299.0	418.0
5		653.0	892.0	389.0	506.0	740.0	1040.0	282.0	417.0
6		752.0	1040.0	348.0	524.0	793.0	1200.0	250.0	334.0
7		731.0	929.0	385.0	533.0	756.0	1000.0	263.0	385.0
8		729.0	978.0	400.0	602.0	851.0	1130.0	310.0	439.0

RAW DATA FOR MANGANESE,MN

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		380.00	532.00	670.00	938.00	736.00	1022.00
LAB/AN	LAB REJECTED						
1		208.00*	285.00	590.00	801.00	777.00	1080.00
2		371.00	531.00	737.00	1010.00	730.00	1000.00
3		335.00	440.00	651.00	874.00	630.00	807.00
4		346.00	474.00	635.00	624.00	646.00	835.00
5		345.00	492.00	654.00	889.00	656.00	881.00
6		288.00	375.00	634.00	886.00	375.00	827.00
7		315.00	477.00	569.00	843.00	585.00	923.00
8		366.00	535.00	757.00	1050.00	724.00	992.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 22 . RAW DATA FOR MOLYBDENUM,MO

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		120.0	150.0	1980.0	2475.0	1020.0	1275.0	600.0	750.0
LAB/AN	LAB REJECTED								
1		195.0	205.0	2830.0	2240.0	1850.0	2490.0	1180.0	1410.0
2		110.0	152.0	1990.0	2570.0	1040.0	1290.0	645.0	807.0
3		135.0	152.0	1630.0	2090.0	1080.0	1310.0	697.0	822.0
4		70.7	70.7	1770.0	2420.0	1010.0	1340.0	623.0	772.0
5		173.0	207.0	3060.0	3530.0*	1640.0	2050.0	1030.0	1260.0
6		109.0	95.0	1940.0	2370.0	933.0	1220.0	608.0	770.0
7		120.0	145.0	1780.0	2000.0	933.0	1140.0	613.0	737.0
8		70.0	58.0	2080.0	2550.0	1130.0	1340.0	667.0	830.0

RAW DATA FOR MOLYBDENUM,MO

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		150.00	188.00	300.00	375.00	3900.00	4875.00
LAB/AN	LAB REJECTED						
1		276.00*	413.00*	444.00	558.00	5650.00	7850.00
2		180.00	226.00	314.00	395.00	3960.00	5100.00
3		188.00	208.00	225.00	210.00	4110.00	4910.00
4		186.00	216.00	325.00	404.00	3920.00	5720.00
5		225.00*	312.00*	402.00	521.00	6440.00	7790.00
6		176.00	220.00	310.00	373.00	3760.00	4540.00
7		170.00	215.00	285.00	360.00	3660.00	3870.00
8		188.00	236.00	318.00	409.00	4000.00	5000.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 23. RAW DATA FOR NICKEL,NI

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		9642.0	12038.0	71.0	99.0	180.0	252.0	530.0	742.0
1		8780.0	10400.0	19.0	17.0	149.0	242.0	473.0	631.0
2		9330.0	12100.0	20.7	27.8	168.0	239.0	502.0	680.0
3		9930.0	11600.0	3.0	15.0	158.0	206.0	456.0	578.0
4		9530.0	12200.0	32.3	63.4	213.0*	306.0	524.0	711.0
5		8900.0	11500.0	42.3	55.6	183.0	253.0	509.0	696.0
6		9820.0	11500.0	23.3	25.6	163.0	203.0	463.0	643.0
7		8830.0	9880.0	21.6	32.4	170.0	216.0	459.0	576.0
8		9620.0	11700.0	15.7	26.1	163.0	221.0	486.0	676.0

RAW DATA FOR NICKEL,NI

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		4836.00	6037.00	43800.00	61300.00	58500.00	81900.00
1		4580.00	5660.00	11700.00*	55800.00	54300.00	74700.00
2		4570.00	5590.00	44200.00	60800.00	57700.00	82800.00
3		5020.00	5770.00	46400.00	59000.00	61200.00	57100.00
4		5080.00	6110.00	46200.00	63300.00	64900.00	86700.00
5		4530.00	5630.00	40500.00	60100.00	56100.00	79700.00
6		4430.00	5370.00	42000.00	57100.00	55000.00	77000.00
7		4330.00	5120.00	35200.00	51200.00	49700.00	63800.00
8		4650.00	5770.00	40500.00	55900.00	52000.00	78500.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 24. RAW DATA FOR POTASSIUM,K

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		13200.0	13500.0	10000.0	14000.0	3670.0	5140.0	2800.0	3920.0
LAB/AN	LAB REJECTED								
1		300.0	450.0	510.0	680.0	350.0	510.0	1830.0	2530.0
2		412.0	593.0	817.0	1120.0	490.0	671.0	2320.0	3090.0
3		550.0	667.0	1060.0	1410.0	626.0	778.0	2010.0	2850.0
4		346.0	502.0	745.0	849.0	398.0	554.0	2300.0	3070.0
5		657.0	712.0	1230.0	1560.0	807.0	1040.0	2380.0	3040.0
6		774.0	1040.0	1550.0	1890.0	879.0	1160.0	3020.0	4150.0
7		965.0	7800.0*	983.0	1280.0	625.0	1200.0	2730.0	3720.0
8		389.0	551.0	723.0	974.0	494.0	557.0	1870.0	2530.0

RAW DATA FOR POTASSIUM,K

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		14600.00	20400.00	900.00	1260.00	750.00	1050.00
LAB/AN	LAB REJECTED						
1		2010.00	2950.00	400.00	540.00	870.00	1260.00
2		3000.00	4500.00	4610.00*	6410.00*	1060.00	1470.00
3		2930.00	3881.00	417.00	555.00	969.00	1220.00
4		2600.00	3660.00	363.00	350.00	1020.00	1400.00
5		3520.00	3540.00	465.00	607.00	1140.00	1660.00
6		3770.00	5490.00	624.00	819.00	1330.00	2140.00
7		3440.00	5020.00	495.00	738.00	1230.00	1840.00
8		2290.00	3700.00	372.00	513.00	913.00	1220.00

* = REJECTED

CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 25. RAW DATA FOR SILVER,AG

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		37.6	46.6	482.1	602.1	187.3	232.3	306.6	381.6
1		51.0	53.0	346.0	395.0	167.0	206.0	264.0	320.0
2		32.2	39.6	363.0	345.0	158.0	100.0*	266.0	306.0
3		31.0	36.0	285.0	379.0	158.0	191.0	412.0*	275.0
4		22.6	25.5	296.0	270.0	155.0	195.0	276.0	329.0
5		56.1	78.0*	318.0	297.0	149.0	196.0	250.0	313.0
6		32.5	37.1	337.0	343.0	159.0	191.0	264.0	331.0
7		46.5	43.0	254.0	360.0	166.0	196.0	246.0	306.0
8		30.3	37.1	353.0	367.0	171.0	188.0	265.0	319.0

RAW DATA FOR SILVER,AG

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		90.00	112.50	105.80	131.30	98.00	137.00
1		84.00	90.00	113.00	119.00	110.00	112.00
2		68.10	75.00	88.10	101.00	90.40	126.00
3		64.00	70.00	91.00	95.00	92.00	124.00
4		58.20	69.60	88.10	101.00	85.20	107.00
5		55.00	62.80	78.60	81.60	75.10	97.50
6		68.20	78.40	91.40	110.00	92.50	136.00
7		76.80	85.70	99.10	115.00	100.00	135.00
8		69.90	76.30	91.90	106.00	93.10	125.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 26 .RAW DATA FOR SODIUM,NA

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		5600.0	7340.0	5100.0	7140.0	3350.0	4690.0	81000.0	113000.0
LAB/AN	LAB REJECTED								
1		1200.0	1500.0	500.0*	500.0	1800.0	2300.0	77100.0	105000.0
2		983.0	1400.0	238.0	328.0	1760.0	2470.0	75700.0	106000.0
3		1090.0	1340.0	320.0	430.0	2060.0	2690.0	83600.0	112000.0
4		924.0	1280.0	262.0	297.0	1780.0	2240.0	79000.0	100000.0
5		1040.0	1570.0	303.0	400.0	1970.0	2810.0	75300.0	107000.0
6		1150.0	1480.0	314.0	448.0	2090.0	2850.0	91700.0*	128000.0*
7		1260.0	1670.0	239.0	374.0	1960.0	3100.0	80100.0	10700.0*
8		1020.0	1340.0	260.0	350.0	1920.0	2470.0	73500.0	104000.0

RAW DATA FOR SODIUM,NA

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		19100.00	26700.00	4720.00	6600.00	54700.00	76600.00
LAB/AN	LAB REJECTED						
1		4300.00	5700.00	4500.00	6100.00	50500.00	72300.00
2		4200.00	5900.00	4590.00	6410.00	52700.00	72400.00
3		4300.00	5640.00	4680.00	6380.00	55200.00	74600.00
4		4300.00	6030.00	4530.00	4820.00	52300.00	72000.00
5		4390.00	101000.00*	4720.00	5490.00	51800.00	68513.00
6		4590.00	6530.00	4990.00	6630.00	62300.00	86700.00
7		4470.00	6520.00	4850.00	6500.00	46900.00	77500.00
8		4170.00	5810.00	4420.00	6100.00	48300.00	67000.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 27 . RAW DATA FOR THALLIUM, TI

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		120.0	150.0	391.0	439.0	210.4	263.0	1952.0	2440.0
LAB/AN	LAB REJECTED								
1		119.0	139.0	355.0	417.0	194.0	257.0	1980.0	2350.0
2		122.0	162.0	400.0	493.0	219.0	267.0	2100.0	2580.0
3		121.0	137.0	369.0	434.0	206.0	234.0	1760.0	2100.0
4		145.0	169.0	392.0	526.0	226.0	288.0	1840.0	2390.0
5		151.0	168.0	427.0	498.0	234.0	290.0	2130.0	2590.0
6		109.0	148.0	417.0	489.0	200.0	246.0	1980.0	2500.0
7		147.0	172.0	397.0	474.0	241.0	276.0	2000.0	2330.0
8		117.0	159.0	401.0	497.0	237.0	264.0	1980.0	2370.0

RAW DATA FOR THALLIUM, TI

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		600.00	750.00	992.00	1240.00	752.00	940.00
LAB/AN	LAB REJECTED						
1		604.00	749.00	934.00	1080.00	672.00	888.00
2		631.00	780.00	986.00	1220.00	764.00	958.00
3		566.00	697.00	825.00	1020.00	693.00	838.00
4		612.00	774.00	957.00	1200.00	740.00	907.00
5		653.00	791.00	1030.00	1280.00	838.00	1010.00
6		647.00	771.00	909.00	1150.00	758.00	889.00
7		629.00	795.00	964.00	1170.00	786.00	893.00
8		652.00	813.00	812.00	1010.00	706.00	844.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 28 . RAW DATA FOR VANADIUM,V

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		1902.0	2371.0	821.0	1009.0	3028.0	3778.0	406.0	500.0
1		1640.0	2070.0	590.0	730.0	2650.0	3470.0	347.0	402.0
2		1930.0	2420.0	795.0	1010.0	3230.0	4000.0	425.0	535.0
3		2310.0	2800.0	969.0	1190.0	3910.0	4810.0	524.0*	641.0
4		1700.0	2180.0	768.0	949.0	2910.0	3630.0	394.0	449.0
5		2320.0	2970.0	862.0	1160.0	4060.0	4690.0	431.0	648.0
6		1860.0	2290.0	772.0	986.0	2960.0	3730.0	408.0	524.0
7		1780.0	2190.0	729.0	871.0	2910.0	3500.0	400.0	486.0
8		1910.0	2320.0	830.0	1030.0	3300.0	4000.0	410.0	510.0

RAW DATA FOR VANADIUM,V

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		330.00	387.00	6006.00	7506.00	4514.00	5633.00
1		256.00	321.00	5490.00	7210.00	4280.00	5250.00
2		345.00	435.00	6420.00	7710.00	4580.00	5960.00
3		384.00	487.00	6950.00	8440.00	5490.00	7130.00
4		380.00	449.00	5580.00	6550.00	4190.00	5580.00
5		316.00	526.00	7680.00	10100.00	5510.00	7450.00
6		342.00	449.00	5980.00	7470.00	4820.00	5480.00
7		329.00	429.00	5360.00	4860.00	4380.00	5000.00
8		320.00	430.00	5720.00	7020.00	4720.00	5650.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 29. RAW DATA FOR ZINC,ZN

LAB/AN	LAB REJECTED	Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		1800.0	2500.0	5220.0	6468.0	1570.0	2200.0	14000.0	19600.0
1		1600.0	2170.0	3900.0	4460.0	1170.0	2020.0	13500.0	16800.0
2		1710.0	2410.0	4900.0	6230.0	1590.0	2250.0	13800.0	19500.0
3		1470.0	2080.0	4430.0	5570.0	1360.0	1910.0	12500.0	17800.0
4		1640.0	2450.0	4620.0	6110.0	1530.0	2180.0	12500.0	18100.0
5		2050.0*	2280.0	5340.0	5450.0	1910.0	2100.0	14100.0	18300.0
6		1730.0	2950.0*	5710.0	6870.0	1540.0	1760.0	12100.0	17100.0
7		1680.0	2240.0	4470.0	5110.0	1520.0	2140.0	11800.0	17600.0
8		1710.0	2270.0	4740.0	5930.0	1550.0	2140.0	12500.0	16600.0

RAW DATA FOR ZINC,ZN

LAB/AN	LAB REJECTED	Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		93.00	130.00	24000.00	33600.00	X60000.00	X24000.00
1		84.00	64.00*	23200.00	31100.00	135000.00	179000.00
2		95.30	129.00	26100.00	36600.00	159000.00	123000.00
3		87.00	107.00	23300.00	31700.00	139000.00	141000.00
4		79.50	127.00	24800.00	33900.00	153000.00	104000.00
5		110.00	122.00	27400.00	34100.00	137000.00	105000.00
6		98.40	135.00	24000.00	32700.00	137000.00	177000.00
7		95.50	131.00	22800.00	28900.00	99600.00	187000.00
8		98.80	136.00	21700.00	33300.00	135000.00	195000.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 30. RAW DATA FOR ARSENIC,AS

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		563.0	686.0	293.0	363.0	106.0	131.0	233.0	237.0
LAB/AN	LAB REJECTED								
1		640.0	680.0	360.0	380.0	120.0	130.0	170.0	180.0
2		449.0	501.0	200.0	245.0	74.8	109.0	125.0	167.0
3		564.0	628.0	324.0	415.0	124.0	168.0	217.0	276.0
4		407.0	508.0	218.0	281.0	144.0	191.0	204.0	228.0
5		446.0	564.0	269.0	292.0	104.0	125.0	208.0	216.0
6		492.0	728.0	740.0*	515.0	249.0*	281.0*	238.0	288.0
7		523.0	672.0	221.0	333.0	85.4	130.0	165.0	247.0
8		516.0	650.0	246.0	351.0	107.0	143.0	209.0	266.0

RAW DATA FOR ARSENIC,AS

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		257.00	307.00	37.00	45.00	25.00	28.00
LAB/AN	LAB REJECTED						
1		320.00	320.00	9.00	10.00	11.00	12.00
2		185.00	210.00	21.20	29.90	16.70	22.20
3		297.00	395.00	36.50	48.30	28.00	40.80
4		255.00	326.00	33.60	45.60	19.80	26.80
5		191.00	262.00	40.10	44.00	17.60	21.80
6		316.00	337.00	31.50	52.70	30.00	58.80
7		267.00	340.00	26.10	32.70	21.80	27.30
8		120.00	334.00	31.30	39.30	22.80	30.80

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 31. RAW DATA FOR SELENIUM,SE

		Solid 1A	Solid 1B	Solid 2A	Solid 2B	Solid 3A	Solid 3B	Solid 4A	Solid 4B
INCREMENT IN UG/G		41.2	32.4	22.0	27.0	3.1	6.4	32.5	40.2
LAB/AN	LAB REJECTED								
1		18.0	14.0	2.6	1.8	0.1	0.1	2.7	3.5
2		20.2	29.2	14.8	15.9	1.5	2.7	17.5	20.9
3		21.4	21.8	17.1	23.5	< 0.3*	< 0.5*	8.8	15.1
4		34.1	33.3	17.2	25.7	6.8	12.0	27.0	35.6
5		26.3	47.4	12.8	23.3	3.2	3.3	23.3	26.5
6		323.0*	381.0*	254.0*	212.0*	91.5*	82.1*	37.1	33.2
7		7.0	7.1	8.5	8.9	0.1	0.1	2.0	2.1
8		37.5	43.1	21.6	25.9	4.4	4.2	25.5	30.3

RAW DATA FOR SELENIUM,SE

		Solid 5A	Solid 5B	Solid 6A	Solid 6B	Solid 7A	Solid 7B
INCREMENT IN UG/G		64.50	76.50	102.00	127.50	81.60	102.00
LAB/AN	LAB REJECTED						
1		21.00	80.00	42.00	100.00	33.00	40.00
2		40.80	52.20	68.10	94.20	57.60	71.00
3		58.20	7.80	73.60	101.00	39.90	51.90
4		55.70	61.30	77.70	83.10	72.30	80.40
5		58.50	75.30	86.00	119.00	66.00	108.00
6		57.60	74.00	64.10	122.00	68.80	83.00
7		33.50	30.10	39.70	44.10	14.40	16.20
8		59.50	70.30	95.00	116.00	78.00	94.50

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

APPENDIX C

STATISTICAL SUMMARIES

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TABLE 32. STATISTICAL SUMMARY FOR ALUMINUM,AL

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	7	8
TRUE CONC (C) UG/G	27400.0	38400.0	1700.00	58400.00
MEAN RECOVERY (X)	4666.3	6260.0	8561.43	13390.00
% RECOVERY	17.03	16.30	20.53	22.93
OVERALL STD DEV (S)	1112.0	1656.0	1069.26	2666.55
OVERALL REL STD DEV, %	23.83	26.45	12.49	19.91
SINGLE STD DEV, (SR)	429.1		901.46	
ANALYST REL DEV, %	7.36		8.21	
3-4 YOUDEN PAIR	**Solid 2A**	**2B**	**Solid 6A**	**6B**
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	42300.0	59200.0	1430.00	2000.00
MEAN RECOVERY (X)	6340.0	8501.3	715.00	905.38
% RECOVERY	14.99	14.36	50.00	45.27
OVERALL STD DEV (S)	1635.0	2136.0	176.48	297.73
OVERALL REL STD DEV, %	25.79	25.13	24.68	32.88
SINGLE STD DEV, (SR)	509.2		106.38	
ANALYST REL DEV, %	6.86		13.13	
5-6 YOUDEN PAIR	**Solid 3A**	**3B**	**Solid 7A**	**7B**
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	25200.0	35300.0	5100.00	7140.00
MEAN RECOVERY (X)	13712.5	18687.5	1344.75	1852.50
% RECOVERY	54.41	52.94	26.37	25.95
OVERALL STD DEV (S)	2202.9	3205.5	296.82	387.22
OVERALL REL STD DEV, %	16.06	17.15	22.07	20.90
SINGLE STD DEV, (SR)	1023.8		137.03	
ANALYST REL DEV, %	6.32		8.57	
7-8 YOUDEN PAIR	**Solid 4A**	**4B**		
NUMBER OF DATA POINTS	8	8		
TRUE CONC (C) UG/G	16900.0	23700.0		
MEAN RECOVERY (X)	6245.0	9028.8		
% RECOVERY	36.95	38.10		
OVERALL STD DEV (S)	2027.5	3059.7		
OVERALL REL STD DEV, %	32.47	33.89		
SINGLE STD DEV, (SR)	851.6			
ANALYST REL DEV, %	11.15			

TABLE 33. STATISTICAL SUMMARY FOR ANTIMONY,SB

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	3937.0	4912.0	603.00	753.00
MEAN RECOVERY (X)	2307.6	2328.3	333.00	350.75
% RECOVERY	58.61	47.40	55.22	46.58
OVERALL STD DEV (S)	1004.0	1374.4	57.15	65.74
OVERALL REL STD DEV, %	43.51	59.03	17.16	18.74
SINGLE STD DEV, (SR)	490.9		18.53	
ANALYST REL DEV, %	21.18		5.42	
3-4 YOUDEN PAIR	**Solid 2A**	**2B**	**Solid 6A**	**6B**
NUMBER OF DATA POINTS	7	7	7	7
TRUE CONC (C) UG/G	221.0	274.0	140.00	196.00
MEAN RECOVERY (X)	116.1	110.1	62.77	91.93
% RECOVERY	52.53	40.18	44.84	46.90
OVERALL STD DEV (S)	12.7	42.4	17.27	23.35
OVERALL REL STD DEV, %	10.94	38.48	27.51	25.40
SINGLE STD DEV, (SR)	30.1		14.95	
ANALYST REL DEV, %	26.63		19.33	
5-6 YOUDEN PAIR	**Solid 3A**	**3B**	**Solid 7A**	**7B**
NUMBER OF DATA POINTS	7	8	7	7
TRUE CONC (C) UG/G	423.0	528.0	920.00	1145.00
MEAN RECOVERY (X)	144.4	177.9	729.29	889.86
% RECOVERY	34.14	33.69	79.27	77.72
OVERALL STD DEV (S)	38.1	40.2	33.88	57.02
OVERALL REL STD DEV, %	26.41	22.59	4.65	6.41
SINGLE STD DEV, (SR)	13.7		25.03	
ANALYST REL DEV, %	11.58		3.09	
7-8 YOUDEN PAIR	**Solid 4A**	**4B**		
NUMBER OF DATA POINTS	8	8		
TRUE CONC (C) UG/G	1935.0	2415.0		
MEAN RECOVERY (X)	1411.3	1700.0		
% RECOVERY	72.93	70.39		
OVERALL STD DEV (S)	214.4	170.9		
OVERALL REL STD DEV, %	15.19	10.05		
SINGLE STD DEV, (SR)	63.3			
ANALYST REL DEV, %	4.07			

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TABLE 34. STATISTICAL SUMMARY FOR BARIUM,AA ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	7	7	5	6
TRUE CONC (C) UG/G	1080.0	1280.0	640.00	896.00
MEAN RECOVERY (X)	735.7	943.7	496.20	642.17
% RECOVERY	68.12	73.73	77.53	71.67
OVERALL STD DEV (S)	61.5	55.3	37.86	187.70
OVERALL REL STD DEV, %	8.36	5.86	7.63	29.23
SINGLE STD DEV, (SR)	53.9		48.47	
ANALYST REL DEV, %	6.42		8.52	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	7	7	7	7
TRUE CONC (C) UG/G	1390.0	1616.0	480.00	672.00
MEAN RECOVERY (X)	1031.9	1371.4	501.29	628.86
% RECOVERY	74.23	84.87	104.43	93.58
OVERALL STD DEV (S)	66.0	98.7	94.43	194.99
OVERALL REL STD DEV, %	6.39	7.20	18.84	31.01
SINGLE STD DEV, (SR)	47.2		83.94	
ANALYST REL DEV, %	3.92		14.85	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	209904.0	212380.0	5252.00	6490.00
MEAN RECOVERY (X)	10346.3	11630.0	2283.25	916.13
% RECOVERY	4.93	5.48	43.47	14.12
OVERALL STD DEV (S)	4965.8	1792.9	1671.48	928.25
OVERALL REL STD DEV, %	48.00	15.42	73.21	101.32
SINGLE STD DEV, (SR)	3325.7		1228.30	
ANALYST REL DEV, %	30.27		76.78	

7-3 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	6
TRUE CONC (C) UG/G	209904.0	292380.0
MEAN RECOVERY (X)	318.4	123.0
% RECOVERY	0.15	0.04
OVERALL STD DEV (S)	282.7	67.1
OVERALL REL STD DEV, %	88.78	54.53
SINGLE STD DEV, (SR)	63.5	
ANALYST REL DEV, %	28.76	

TABLE 35 . STATISTICAL SUMMARY FOR BERYLLIUM/BE ANALYSES

1-2 YOUDEN PAIR		Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS		7	8	8	8
TRUE CONC (C) UG/G		19.2	24.0	4.80	6.00
MEAN RECOVERY (X)		18.5	22.7	5.48	6.55
% RECOVERY		96.50	94.64	114.19	109.23
OVERALL STD DEV (S)		0.9	1.0	0.91	0.69
OVERALL REL STD DEV, %		5.00	4.60	16.53	10.52
SINGLE	STD DEV, (SR)	0.6		0.53	
ANALYST	REL DEV, %	3.00		8.74	
3-4 YOUDEN PAIR		Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS		8	8	8	8
TRUE CONC (C) UG/G		38.4	48.0	96.00	120.00
MEAN RECOVERY (X)		35.5	44.8	89.69	111.13
% RECOVERY		92.42	93.41	93.42	92.60
OVERALL STD DEV (S)		3.9	2.5	6.81	9.52
OVERALL REL STD DEV, %		10.89	5.64	7.59	8.57
SINGLE	STD DEV, (SR)	3.2		3.53	
ANALYST	REL DEV, %	7.87		3.51	
5-6 YOUDEN PAIR		Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS		8	8	8	8
TRUE CONC (C) UG/G		198.4	243.0	80.00	100.00
MEAN RECOVERY (X)		188.0	231.0	78.65	97.19
% RECOVERY		94.76	93.15	98.31	97.19
OVERALL STD DEV (S)		13.8	9.3	6.22	7.69
OVERALL REL STD DEV, %		7.34	4.04	7.90	7.91
SINGLE	STD DEV, (SR)	7.7		3.06	
ANALYST	REL DEV, %	3.67		3.48	
7-8 YOUDEN PAIR		Solid 4A	4B		
NUMBER OF DATA POINTS		8	8		
TRUE CONC (C) UG/G		144.0	180.0		
MEAN RECOVERY (X)		145.4	172.5		
% RECOVERY		100.95	95.33		
OVERALL STD DEV (S)		14.9	14.4		
OVERALL REL STD DEV, %		10.28	8.32		
SINGLE	STD DEV, (SR)	8.3			
ANALYST	REL DEV, %	5.51			

TABLE 36. STATISTICAL SUMMARY FOR CADMIUM,CD ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	10.2	14.3	74.40	93.00
MEAN RECOVERY (X)	9.0	11.7	72.92	91.19
% RECOVERY	88.37	82.17	98.02	98.05
OVERALL STD DEV (S)	2.8	2.7	4.07	4.38
OVERALL REL STD DEV, %	30.94	22.85	5.58	4.80
SINGLE STD DEV, (SR)	0.9		0.98	
ANALYST REL DEV, %	8.57		1.20	
3-4 YOUDEN PAIR	**Solid 2A**	**2B**	**Solid 6A**	**6B**
NUMBER OF DATA POINTS	7	8	8	8
TRUE CONC (C) UG/G	59.6	74.5	12500.00	17500.00
MEAN RECOVERY (X)	60.6	67.9	11750.00	17212.50
% RECOVERY	101.61	91.16	94.00	98.36
OVERALL STD DEV (S)	1.8	10.5	707.11	875.73
OVERALL REL STD DEV, %	3.03	15.40	6.02	5.09
SINGLE STD DEV, (SR)	7.1		550.24	
ANALYST REL DEV, %	11.05		3.80	
5-6 YOUDEN PAIR	**Solid 3A**	**3B**	**Solid 7A**	**7B**
NUMBER OF DATA POINTS	7	7	7	7
TRUE CONC (C) UG/G	20.0	28.0	5.96	7.45
MEAN RECOVERY (X)	21.8	30.5	5.58	6.13
% RECOVERY	109.21	108.93	93.65	82.30
OVERALL STD DEV (S)	1.4	1.9	2.88	3.83
OVERALL REL STD DEV, %	6.40	6.22	51.54	62.42
SINGLE STD DEV, (SR)	1.1		1.79	
ANALYST REL DEV, %	4.08		30.49	
7-8 YOUDEN PAIR	**Solid 4A**	**4B**		
NUMBER OF DATA POINTS	7	7		
TRUE CONC (C) UG/G	110.0	154.0		
MEAN RECOVERY (X)	109.0	154.7		
% RECOVERY	99.10	100.46		
OVERALL STD DEV (S)	7.5	8.7		
OVERALL REL STD DEV, %	6.86	5.59		
SINGLE STD DEV, (SR)	5.4			
ANALYST REL DEV, %	4.08			

TABLE 37. STATISTICAL SUMMARY FOR CALCIUM, CA ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	7	7	8	7
TRUE CONC (C) UG/G	28900.0	40500.0	140000.00	196000.00
MEAN RECOVERY (X)	28042.9	39128.6	141125.00	195000.00
% RECOVERY	97.03	96.61	100.80	99.49
OVERALL STD DEV (S)	1202.5	2339.9	8626.03	18999.94
OVERALL REL STD DEV, %	4.29	6.03	6.11	9.74
SINGLE STD DEV, (SR)	1058.6		9939.09	
ANALYST REL DEV, %	3.15		5.91	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	7	7
TRUE CONC (C) UG/G	75000.0	105000.0	69200.00	96900.00
MEAN RECOVERY (X)	72437.5	101125.0	70542.81	99128.56
% RECOVERY	96.58	96.31	101.94	102.30
OVERALL STD DEV (S)	4954.4	9555.7	5695.41	7569.07
OVERALL REL STD DEV, %	6.84	9.45	8.07	7.64
SINGLE STD DEV, (SR)	7039.[illegible]		2729.27	
ANALYST REL DEV, %	8.11		3.22	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	68000.0	95200.0	14900.00	20900.00
MEAN RECOVERY (X)	65587.5	91462.5	11938.75	15527.50
% RECOVERY	96.45	96.07	80.13	74.29
OVERALL STD DEV (S)	5389.8	8138.4	3337.76	5216.81
OVERALL REL STD DEV, %	8.22	8.90	27.96	33.60
SINGLE STD DEV, (SR)	2691.2		1837.29	
ANALYST REL DEV, %	3.43		13.38	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	53000.0	74200.0
MEAN RECOVERY (X)	48350.0	66337.5
% RECOVERY	91.23	89.40
OVERALL STD DEV (S)	4697.1	9655.1
OVERALL REL STD DEV, %	9.71	14.55
SINGLE STD DEV, (SR)	4614.8	
ANALYST REL DEV, %	8.05	

TABLE 38. STATISTICAL SUMMARY FOR CHROMIUM,CR ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	28700.0	40100.0	1004.00	1405.00
MEAN RECOVERY (X)	28290.0	40312.5	1102.50	1362.50
% RECOVERY	98.57	100.53	109.81	96.98
OVERALL STD DEV (S)	3144.1	2828.6	95.13	135.73
OVERALL REL STD DEV, %	11.11	7.02	8.63	9.96
SINGLE STD DEV, (SR)	1452.6		41.06	
ANALYST REL DEV, %	4.23		3.33	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	3	8	5	8
TRUE CONC (C) UG/G	180.0	252.0	66000.00	92400.00
MEAN RECOVERY (X)	129.3	178.0	72340.00	97675.00
% RECOVERY	71.31	70.63	109.61	105.71
OVERALL STD DEV (S)	24.8	28.8	336.02	4439.87
OVERALL REL STD DEV, %	19.17	16.16	0.46	4.55
SINGLE STD DEV, (SR)	6.3		3065.70	
ANALYST REL DEV, %	4.12		3.61	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	7	8
TRUE CONC (C) UG/G	92.0	129.0	80000.00	112000.00
MEAN RECOVERY (X)	67.5	91.7	79528.56	111225.00
% RECOVERY	73.36	71.09	99.41	99.31
OVERALL STD DEV (S)	11.0	16.4	10370.65	13234.29
OVERALL REL STD DEV, %	16.32	17.93	13.04	11.90
SINGLE STD DEV, (SR)	6.1		10868.86	
ANALYST REL DEV, %	7.66		11.40	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	8100.0	11332.0
MEAN RECOVERY (X)	8507.5	11900.0
% RECOVERY	105.03	105.01
OVERALL STD DEV (S)	353.7	621.1
OVERALL REL STD DEV, %	4.16	5.22
SINGLE STD DEV, (SR)	456.4	
ANALYST REL DEV, %	4.47	

TABLE 39. STATISTICAL SUMMARY FOR COBALT,CO ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	6
TRUE CONC (C) UG/G	288.2	358.2	90.80	110.80
MEAN RECOVERY (X)	294.9	363.0	93.95	109.17
% RECOVERY	102.32	101.34	103.47	98.53
OVERALL STD DEV (S)	20.7	26.3	16.18	5.42
OVERALL REL STD DEV, %	7.03	7.24	17.22	4.96
SINGLE STD DEV, (SR)	6.0		1.97	
ANALYST REL DEV, %	1.83		1.94	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	112.0	136.0	417.00	517.00
MEAN RECOVERY (X)	107.3	130.3	414.00	537.25
% RECOVERY	95.76	95.77	99.28	103.92
OVERALL STD DEV (S)	14.9	23.7	46.14	104.25
OVERALL REL STD DEV, %	13.93	18.19	11.15	19.41
SINGLE STD DEV, (SR)	7.7		54.77	
ANALYST REL DEV, %	6.45		11.52	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	7	7	7	6
TRUE CONC (C) UG/G	513.0	637.0	87.00	121.00
MEAN RECOVERY (X)	513.6	624.0	90.70	118.83
% RECOVERY	100.11	97.96	104.25	98.21
OVERALL STD DEV (S)	34.3	41.3	12.93	3.37
OVERALL REL STD DEV, %	6.68	6.61	14.26	2.84
SINGLE STD DEV, (SR)	42.2		5.23	
ANALYST REL DEV, %	7.43		4.99	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	204.0	254.4
MEAN RECOVERY (X)	211.1	254.5
% RECOVERY	103.49	100.04
OVERALL STD DEV (S)	19.7	22.1
OVERALL REL STD DEV, %	9.31	8.68
SINGLE STD DEV, (SR)	3.5	
ANALYST REL DEV, %	1.52	

TABLE 40. STATISTICAL SUMMARY FOR COPPER,CU ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	7	7
TRUE CONC (C) UG/G	120.0	168.0	105.00	147.00
MEAN RECOVERY (X)	143.9	218.4	66.09	80.54
% RECOVERY	119.90	129.99	62.94	54.79
OVERALL STD DEV (S)	5.4	16.4	3.31	7.67
OVERALL REL STD DEV, %	3.74	7.51	5.01	9.52
SINGLE ANALYST STD DEV, (SR)	10.7		5.41	
SINGLE ANALYST REL DEV, %	5.89		7.38	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	5004.0	6205.0	18600.00	25900.00
MEAN RECOVERY (X)	5056.3	6113.8	18612.50	25837.50
% RECOVERY	101.04	98.53	100.07	99.76
OVERALL STD DEV (S)	517.8	521.3	941.77	1270.47
OVERALL REL STD DEV, %	10.24	8.53	5.06	4.92
SINGLE ANALYST STD DEV, (SR)	142.1		863.34	
SINGLE ANALYST REL DEV, %	2.54		3.88	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	7	6
TRUE CONC (C) UG/G	450.0	630.0	2700.00	3780.00
MEAN RECOVERY (X)	365.3	568.3	1532.86	2181.25
% RECOVERY	81.17	90.20	56.77	57.71
OVERALL STD DEV (S)	20.6	15.8	84.20	390.07
OVERALL REL STD DEV, %	5.64	2.78	5.49	17.88
SINGLE ANALYST STD DEV, (SR)	13.7		182.69	
SINGLE ANALYST REL DEV, %	2.93		9.84	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	810.0	1130.0
MEAN RECOVERY (X)	819.1	1137.5
% RECOVERY	101.13	100.66
OVERALL STD DEV (S)	51.8	99.8
OVERALL REL STD DEV, %	6.33	8.79
SINGLE ANALYST STD DEV, (SR)	41.4	
SINGLE ANALYST REL DEV, %	4.24	

TABLE 41.STATISTICAL SUMMARY FOR IRON,FE ANALYSES

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----------------------------------------------------------------------------------------------------
1-2 YOUDEN PAIR          Solid  1A         1B           Solid  5A        5B
NUMBER OF DATA POINTS           8          8                   8         7
TRUE CONC (C) UG/G       107000.0 150000.0              23900.00  33500.00
MEAN RECOVERY (X)         89212.5 123375.0              20350.00  28757.14
% RECOVERY                  83.38    82.25                 85.15     85.84
OVERALL STD DEV (S)        5306.3   6926.9               1374.23   2712.22
OVERALL REL STD DEV, %       6.51     5.61                  6.75      9.43

SINGLE    STD DEV, (SR)        3407.4                          1629.64
ANALYST   REL DEV, %              3.21                            6.64
----------------------------------------------------------------------------------------------------
3-4 YOUDEN PAIR          Solid  2A         2B           Solid  6A        6B
NUMBER OF DATA POINTS           8          8                   8         7
TRUE CONC (C) UG/G        37000.0  51800.0              59100.00  82700.00
MEAN RECOVERY (X)         20437.5  26137.5              59700.00  83100.00
% RECOVERY                  55.24    50.46                101.02    100.48
OVERALL STD DEV (S)        2002.1   2572.3               2961.65   5541.23
OVERALL REL STD DEV, %       9.80     9.84                  4.96      6.67

SINGLE    STD DEV, (SR)         813.3                          2958.24
ANALYST   REL DEV, %              3.49                            4.14
----------------------------------------------------------------------------------------------------
5-6 YOUDEN PAIR          Solid  3A         3B           Solid  7A        7B
NUMBER OF DATA POINTS           8          8                   8         8
TRUE CONC (C) UG/G        19100.0  26700.0              85000.00119000.00
MEAN RECOVERY (X)         16862.5  23437.5              78362.50110887.50
% RECOVERY                  88.29    87.78                 92.19     93.18
OVERALL STD DEV (S)        1470.5   1672.4               4335.40   6027.25
OVERALL REL STD DEV, %       8.72     7.14                  5.53      5.44

SINGLE    STD DEV, (SR)         650.0                          3459.81
ANALYST   REL DEV, %              3.23                            3.66
----------------------------------------------------------------------------------------------------
7-8 YOUDEN PAIR          Solid  4A         4B
NUMBER OF DATA POINTS           8          8
TRUE CONC (C) UG/G        38100.0  53300.0
MEAN RECOVERY (X)         33662.5  46587.5
% RECOVERY                  88.35    87.41
OVERALL STD DEV (S)        2427.2   3157.5
OVERALL REL STD DEV, %       7.21     6.78

SINGLE    STD DEV, (SR)        1694.0
ANALYST   REL DEV, %              4.22
----------------------------------------------------------------------------------------------------
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TABLE 42 . STATISTICAL SUMMARY FOR LEAD,PB ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	7	6
TRUE CONC (C) UG/G	730.0	1020.0	5033.00	6292.00
MEAN RECOVERY (X)	715.5	971.9	5092.86	6258.33
% RECOVERY	98.01	95.28	101.19	99.46
OVERALL STD DEV (S)	23.9	51.2	148.29	56.34
OVERALL REL STD DEV, %	3.34	5.27	2.91	0.90
SINGLE STD DEV, (SR)	24.1		116.93	
ANALYST REL DEV, %	2.86		2.06	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	7	8	8
TRUE CONC (C) UG/G	230.0	320.0	1020.00	1430.00
MEAN RECOVERY (X)	229.9	317.9	1076.25	1520.00
% RECOVERY	99.95	99.33	105.51	106.29
OVERALL STD DEV (S)	34.6	25.4	44.06	60.47
OVERALL REL STD DEV, %	15.07	7.98	4.09	3.98
SINGLE STD DEV, (SR)	17.5		31.61	
ANALYST REL DEV, %	6.40		2.43	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	15200.0	21300.0	930.00	1300.00
MEAN RECOVERY (X)	15250.0	20787.5	1056.00	1466.25
% RECOVERY	100.33	97.59	113.55	112.79
OVERALL STD DEV (S)	900.8	1324.9	51.07	88.63
OVERALL REL STD DEV, %	5.91	6.37	4.84	6.04
SINGLE STD DEV, (SR)	482.5		63.70	
ANALYST REL DEV, %	2.68		5.05	

7-8 YOUDEN PAIR	Solid 4A	4B,
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	1180.0	1650.0
MEAN RECOVERY (X)	1232.5	1597.5
% RECOVERY	104.45	96.82
OVERALL STD DEV (S)	54.7	63.0
OVERALL REL STD DEV, %	4.44	3.94
SINGLE STD DEV, (SR)	43.9	
ANALYST REL DEV, %	3.10	

TABLE 43 . STATISTICAL SUMMARY FOR MAGNESIUM/MG ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	7	8	8	7
TRUE CONC (C) UG/G	8400.0	11800.0	43000.00	60200.00
MEAN RECOVERY (X)	6797.1	9117.5	37587.50	50685.71
% RECOVERY	80.92	77.27	87.41	84.20
OVERALL STD DEV (S)	605.8	1041.6	2109.44	3657.60
OVERALL REL STD DEV, %	8.91	11.42	5.61	7.22
SINGLE STD DEV, (SR)	797.6		1609.71	
ANALYST REL DEV, %	10.02		3.65	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	7
TRUE CONC (C) UG/G	32000.0	44800.0	19200.00	26900.00
MEAN RECOVERY (X)	25437.5	34687.5	17125.00	24485.71
% RECOVERY	79.49	77.43	89.19	91.02
OVERALL STD DEV (S)	916.4	1642.7	820.67	1290.23
OVERALL REL STD DEV, %	3.60	4.74	4.79	5.27
SINGLE STD DEV, (SR)	851.9		373.84	
ANALYST REL DEV, %	2.83		1.80	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	2300.0	3220.0	1900.00	2660.00
MEAN RECOVERY (X)	2436.3	3338.8	2317.50	3140.00
% RECOVERY	105.92	105.24	121.97	118.05
OVERALL STD DEV (S)	225.6	263.7	196.23	326.58
OVERALL REL STD DEV, %	9.26	7.93	8.47	10.40
SINGLE STD DEV, (SR)	143.3		125.34	
ANALYST REL DEV, %	4.92		4.59	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	2000.0	2800.0
MEAN RECOVERY (X)	2050.0	2912.5
% RECOVERY	102.50	104.02
OVERALL STD DEV (S)	221.2	326.0
OVERALL REL STD DEV, %	10.79	11.19
SINGLE STD DEV, (SR)	107.2	
ANALYST REL DEV, %	4.32	

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TABLE 44 . STATISTICAL SUMMARY FOR MANGANESE,MN ANALYSES

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----------------------------------------------------------------------------------------------------------
1-2 YOUDEN PAIR              Solid  1A        1B          Solid  5A         5B
NUMBER OF DATA POINTS               8         8                  7          8
TRUE CONC (C) UG/G              750.0    1050.0              380.00     532.00
MEAN RECOVERY (X)               725.0     998.1              338.00     451.13
% RECOVERY                      96.67     95.06               88.95      84.80
OVERALL STD DEV (S)              39.6      83.9               28.94      84.34
OVERALL REL STD DEV, %           5.46      8.40                8.56      18.70

SINGLE    STD DEV, (SR)              37.0
ANALYST   REL DEV, %                 4.30                       22.14
                                                                 5.61
----------------------------------------------------------------------------------------------------------
3-4 YOUDEN PAIR              Solid  2A        2B          Solid  6A         6B
NUMBER OF DATA POINTS               7         7                  8          8
TRUE CONC (C) UG/G              550.0     770.0              670.00     938.00
MEAN RECOVERY (X)               396.0     553.4              653.38     872.13
% RECOVERY                      72.00     71.37               97.52      92.98
OVERALL STD DEV (S)              25.5      27.8               65.01     130.17
OVERALL REL STD DEV, %           6.45      5.02                9.95      14.93

SINGLE    STD DEV, (SR)              21.8
ANALYST   REL DEV, %                 4.60                       68.53
                                                                 8.98
----------------------------------------------------------------------------------------------------------
5-6 YOUDEN PAIR              Solid  3A        3B          Solid  7A         7B
NUMBER OF DATA POINTS               7         7                  8          8
TRUE CONC (C) UG/G              810.0    1130.0              736.00    1022.00
MEAN RECOVERY (X)               794.0    1105.7              665.38     918.13
% RECOVERY                      98.02     97.85               90.40      89.84
OVERALL STD DEV (S)              44.3      68.3               72.13      98.07
OVERALL REL STD DEV, %           5.58      6.22               10.84      10.68

SINGLE    STD DEV, (SR)              36.7
ANALYST   REL DEV, %                 3.87                       38.63
                                                                 4.88
----------------------------------------------------------------------------------------------------------
7-8 YOUDEN PAIR              Solid  4A        4B
NUMBER OF DATA POINTS               8         8
TRUE CONC (C) UG/G              330.0     460.0
MEAN RECOVERY (X)               286.6     402.5
% RECOVERY                      86.86     87.50
OVERALL STD DEV (S)              21.3      32.8
OVERALL REL STD DEV, %           7.43      8.15

SINGLE    STD DEV, (SR)              11.5
ANALYST   REL DEV, %                 3.35
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TABLE 45. STATISTICAL SUMMARY FOR MOLYBDENUM,MO ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	6	6
TRUE CONC (C) UG/G	120.0	150.0	150.00	188.00
MEAN RECOVERY (X)	122.3	135.6	181.33	220.17
% RECOVERY	102.36	90.39	120.89	117.11
OVERALL STD DEV (S)	44.3	56.6	7.34	9.77
OVERALL REL STD DEV, %	36.08	41.74	4.05	4.44
SINGLE ANALYST STD DEV, (SR)	14.6		7.96	
SINGLE ANALYST REL DEV, %	11.28		3.96	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	7	8	8
TRUE CONC (C) UG/G	1980.0	2475.0	300.00	375.00
MEAN RECOVERY (X)	2141.3	2320.0	327.88	403.75
% RECOVERY	108.14	93.74	109.29	107.67
OVERALL STD DEV (S)	516.4	219.7	67.63	105.59
OVERALL REL STD DEV, %	24.12	9.47	20.63	26.15
SINGLE ANALYST STD DEV, (SR)	296.7		29.28	
SINGLE ANALYST REL DEV, %	13.30		8.00	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	1020.0	1275.0	3900.00	4875.00
MEAN RECOVERY (X)	1202.0	1522.5	4437.50	5597.50
% RECOVERY	117.84	119.41	113.78	114.82
OVERALL STD DEV (S)	346.3	480.8	1023.87	1467.53
OVERALL REL STD DEV, %	23.31	31.58	23.07	26.22
SINGLE ANALYST STD DEV, (SR)	103.4		441.57	
SINGLE ANALYST REL DEV, %	7.59		8.80	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	600.0	750.0
MEAN RECOVERY (X)	756.6	926.0
% RECOVERY	126.10	123.47
OVERALL STD DEV (S)	220.4	257.4
OVERALL REL STD DEV, %	29.12	27.80
SINGLE ANALYST STD DEV, (SR)	28.2	
SINGLE ANALYST REL DEV, %	3.36	

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TABLE 46 . STATISTICAL SUMMARY FOR NICKEL,NI ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	9642.0	12038.0	4836.00	6037.00
MEAN RECOVERY (X)	9342.5	11360.0	4648.75	5627.50
% RECOVERY	96.89	94.37	96.13	93.22
OVERALL STD DEV (S)	456.7	808.7	266.80	292.70
OVERALL REL STD DEV, %	4.89	7.12	5.74	5.20
SINGLE STD DEV, (SR)	435.2		99.53	
ANALYST REL DEV, %	4.21		1.94	
3-4 YOUDEN PAIR	**Solid 2A**	**2B**	**Solid 6A**	**6B**
NUMBER OF DATA POINTS	8	8	7	8
TRUE CONC (C) UG/G	71.0	99.0	43800.00	61300.00
MEAN RECOVERY (X)	22.2	32.9	42142.86	57900.00
% RECOVERY	31.32	33.19	96.22	94.45
OVERALL STD DEV (S)	11.5	17.5	3925.06	3733.24
OVERALL REL STD DEV, %	51.86	53.26	9.31	6.45
SINGLE STD DEV, (SR)	6.9		1506.56	
ANALYST REL DEV, %	25.10		3.01	
5-6 YOUDEN PAIR	**Solid 3A**	**3B**	**Solid 7A**	**7B**
NUMBER OF DATA POINTS	7	8	8	8
TRUE CONC (C) UG/G	180.0	252.0	58500.00	81900.00
MEAN RECOVERY (X)	164.9	235.8	56362.50	75037.50
% RECOVERY	91.59	93.55	96.35	91.62
OVERALL STD DEV (S)	10.6	33.5	4894.29	9870.50
OVERALL REL STD DEV, %	6.41	14.20	8.68	13.15
SINGLE STD DEV, (SR)	13.1		7021.37	
ANALYST REL DEV, %	6.52		10.69	
7-8 YOUDEN PAIR	**Solid 4A**	**4B**		
NUMBER OF DATA POINTS	8	8		
TRUE CONC (C) UG/G	530.0	742.0		
MEAN RECOVERY (X)	484.0	648.9		
% RECOVERY	91.32	87.45		
OVERALL STD DEV (S)	25.4	51.4		
OVERALL REL STD DEV, %	5.25	7.92		
SINGLE STD DEV, (SR)	21.0			
ANALYST REL DEV, %	3.71			

TABLE 47 . STATISTICAL SUMMARY FOR POTASSIUM,K ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	7	8	8
TRUE CONC (C) UG/G	13200.0	18500.0	14600.00	20400.00
MEAN RECOVERY (X)	549.1	645.0	2945.00	4092.63
% RECOVERY	4.16	3.49	20.17	20.06
OVERALL STD DEV (S)	233.9	196.2	618.96	843.10
OVERALL REL STD DEV, %	42.59	30.42	21.02	20.60
SINGLE STD DEV, (SR)	45.2		384.39	
ANALYST REL DEV, %	7.57		10.92	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	7	7
TRUE CONC (C) UG/G	10000.0	14000.0	900.00	1260.00
MEAN RECOVERY (X)	952.3	1220.4	448.00	593.14
% RECOVERY	9.52	8.72	49.78	47.07
OVERALL STD DEV (S)	328.7	397.1	91.05	146.24
OVERALL REL STD DEV, %	34.52	32.54	20.32	24.66
SINGLE STD DEV, (SR)	62.5		48.81	
ANALYST REL DEV, %	5.75		9.38	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	3670.0	5140.0	750.00	1050.00
MEAN RECOVERY (X)	583.6	808.8	1066.50	1526.25
% RECOVERY	15.90	15.73	142.20	145.36
OVERALL STD DEV (S)	187.6	234.7	158.32	331.79
OVERALL REL STD DEV, %	32.15	35.20	14.84	21.74
SINGLE STD DEV, (SR)	109.6		128.03	
ANALYST REL DEV, %	15.74		9.88	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	2800.0	3920.0
MEAN RECOVERY (X)	2307.5	3122.5
% RECOVERY	82.41	79.66
OVERALL STD DEV (S)	414.0	560.6
OVERALL REL STD DEV, %	17.94	17.95
SINGLE STD DEV, (SR)	113.3	
ANALYST REL DEV, %	4.36	

TABLE 48. STATISTICAL SUMMARY FOR SILVER,AG ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	7	8	8
TRUE CONC (C) UG/G	37.6	46.6	90.00	112.50
MEAN RECOVERY (X)	37.8	38.8	68.02	75.97
% RECOVERY	100.50	83.17	75.58	67.53
OVERALL STD DEV (S)	11.8	8.3	9.40	8.85
OVERALL REL STD DEV, %	31.26	21.34	13.81	11.65
SINGLE STD DEV, (SR)	2.6		1.44	
ANALYST REL DEV, %	6.78		2.00	
3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	482.1	602.1	105.80	131.30
MEAN RECOVERY (X)	319.0	344.5	92.65	103.57
% RECOVERY	66.17	57.22	87.57	78.88
OVERALL STD DEV (S)	37.9	41.9	10.00	11.86
OVERALL REL STD DEV, %	11.88	12.17	10.79	11.45
SINGLE STD DEV, (SR)	36.8		4.11	
ANALYST REL DEV, %	11.09		4.19	
5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	7	8	8
TRUE CONC (C) UG/G	137.3	232.3	98.00	137.00
MEAN RECOVERY (X)	160.4	194.7	92.29	120.31
% RECOVERY	85.62	83.82	94.17	87.82
OVERALL STD DEV (S)	7.2	5.8	10.16	13.61
OVERALL REL STD DEV, %	4.47	2.99	11.01	11.31
SINGLE STD DEV, (SR)	6.7		8.96	
ANALYST REL DEV, %	3.78		8.43	
7-8 YOUDEN PAIR	Solid 4A	4B		
NUMBER OF DATA POINTS	7	8		
TRUE CONC (C) UG/G	306.6	381.6		
MEAN RECOVERY (X)	261.6	312.3		
% RECOVERY	85.31	81.83		
OVERALL STD DEV (S)	10.2	17.7		
OVERALL REL STD DEV, %	3.91	5.67		
SINGLE STD DEV, (SR)	6.2			
ANALYST REL DEV, %	2.16			

TABLE 49 . STATISTICAL SUMMARY FOR SODIUM,NA ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	7
TRUE CONC (C) UG/G	5600.0	7840.0	19100.00	26700.00
MEAN RECOVERY (X)	1083.4	1447.5	4340.00	6018.57
% RECOVERY	19.35	18.46	22.72	22.54
OVERALL STD DEV (S)	113.8	131.9	138.97	368.66
OVERALL REL STD DEV, %	10.50	9.11	3.20	6.13
SINGLE ANALYST STD DEV, (SR)	61.4		183.20	
SINGLE ANALYST REL DEV, %	4.35		3.54	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	7	8	8	8
TRUE CONC (C) UG/G	5100.0	7140.0	4720.00	6600.00
MEAN RECOVERY (X)	276.6	390.9	4660.00	6053.75
% RECOVERY	5.42	5.47	98.73	91.72
OVERALL STD DEV (S)	35.1	67.1	190.48	610.48
OVERALL REL STD DEV, %	12.67	17.16	4.09	10.08
SINGLE ANALYST STD DEV, (SR)	24.0		390.38	
SINGLE ANALYST REL DEV, %	7.19		7.29	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	3350.0	4690.0	54700.00	76600.00
MEAN RECOVERY (X)	1917.5	2616.3	52500.00	73876.63
% RECOVERY	57.24	55.78	95.98	96.44
OVERALL STD DEV (S)	126.6	296.6	4734.36	6121.62
OVERALL REL STD DEV, %	6.60	11.34	9.02	8.29
SINGLE ANALYST STD DEV, (SR)	156.3		3080.27	
SINGLE ANALYST REL DEV, %	6.39		4.87	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	7	6
TRUE CONC (C) UG/G	81000.0	113000.0
MEAN RECOVERY (X)	78471.4	105666.6
% RECOVERY	96.88	93.51
OVERALL STD DEV (S)	2853.0	3932.7
OVERALL REL STD DEV, %	3.64	3.72
SINGLE ANALYST STD DEV, (SR)	2695.1	
SINGLE ANALYST REL DEV, %	2.93	

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TABLE 50 . STATISTICAL SUMMARY FOR THALLIUM/TI ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	120.0	150.0	600.00	750.00
MEAN RECOVERY (X)	128.9	159.3	624.25	771.25
% RECOVERY	107.40	106.17	104.04	102.83
OVERALL STD DEV (S)	16.1	17.4	29.61	35.44
OVERALL REL STD DEV, %	12.51	10.94	4.74	4.59
SINGLE ANALYST STD DEV, (SR)	7.2		10.88	
SINGLE ANALYST REL DEV, %	5.01		1.56	
3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	391.0	439.0	992.00	1240.00
MEAN RECOVERY (X)	395.5	473.5	927.13	1141.25
% RECOVERY	101.15	109.00	93.46	92.04
OVERALL STD DEV (S)	23.5	36.0	75.93	96.72
OVERALL REL STD DEV, %	5.93	7.53	8.19	8.48
SINGLE ANALYST STD DEV, (SR)	15.5		24.74	
SINGLE ANALYST REL DEV, %	3.55		2.39	
5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	210.4	263.0	752.00	940.00
MEAN RECOVERY (X)	219.6	265.3	744.63	903.38
% RECOVERY	104.38	100.86	99.02	96.10
OVERALL STD DEV (S)	17.9	19.5	54.03	56.97
OVERALL REL STD DEV, %	8.13	7.36	7.26	6.31
SINGLE ANALYST STD DEV, (SR)	10.2		25.12	
SINGLE ANALYST REL DEV, %	4.20		3.05	
7-8 YOUDEN PAIR	Solid 4A	4B		
NUMBER OF DATA POINTS	8	8		
TRUE CONC (C) UG/G	1952.0	2440.0		
MEAN RECOVERY (X)	1971.3	2401.3		
% RECOVERY	100.99	98.41		
OVERALL STD DEV (S)	122.3	159.0		
OVERALL REL STD DEV, %	6.20	6.62		
SINGLE ANALYST STD DEV, (SR)	59.3			
SINGLE ANALYST REL DEV, %	2.71			

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TABLE 51. STATISTICAL SUMMARY FOR VANADIUM/V ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	1902.0	2371.0	330.00	387.00
MEAN RECOVERY (X)	1931.3	2405.0	334.00	440.75
% RECOVERY	101.54	101.43	101.21	113.89
OVERALL STD DEV (S)	256.7	317.4	40.41	58.76
OVERALL REL STD DEV, %	13.29	13.20	12.10	13.33
SINGLE STD DEV, (SR)	55.8		31.82	
ANALYST REL DEV, %	2.57		8.22	
3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	821.0	1009.0	6006.00	7506.00
MEAN RECOVERY (X)	789.4	990.8	6147.50	7420.00
% RECOVERY	96.15	98.19	102.36	98.85
OVERALL STD DEV (S)	109.1	148.5	815.22	1503.52
OVERALL REL STD DEV, %	13.83	14.99	13.26	20.26
SINGLE STD DEV, (SR)	35.6		588.34	
ANALYST REL DEV, %	4.00		8.67	
5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	3028.0	3778.0	4514.00	5633.00
MEAN RECOVERY (X)	3241.3	3978.8	4746.25	5937.50
% RECOVERY	107.04	105.31	105.15	105.41
OVERALL STD DEV (S)	502.7	516.8	511.46	884.92
OVERALL REL STD DEV, %	15.51	12.99	10.78	14.90
SINGLE STD DEV, (SR)	70.9		334.12	
ANALYST REL DEV, %	1.96		6.25	
7-8 YOUDEN PAIR	Solid 4A	4B		
NUMBER OF DATA POINTS	7	8		
TRUE CONC (C) UG/G	406.0	500.0		
MEAN RECOVERY (X)	402.1	524.4		
% RECOVERY	99.05	104.87		
OVERALL STD DEV (S)	27.6	85.6		
OVERALL REL STD DEV, %	6.86	16.33		
SINGLE STD DEV, (SR)	38.8			
ANALYST REL DEV, %	8.38			

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TABLE 52. STATISTICAL SUMMARY FOR ZINC/ZN ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1R	Solid 5A	5B
NUMBER OF DATA POINTS	7	7	8	7
TRUE CONC (C) UG/G	1800.0	2500.0	93.00	130.00
MEAN RECOVERY (X)	1648.6	2271.4	93.56	126.71
% RECOVERY	91.59	90.86	100.60	97.47
OVERALL STD DEV (S)	90.8	128.5	9.71	9.91
OVERALL REL STD DEV, %	5.51	5.66	10.38	7.82
SINGLE ANALYST STD DEV, (SR)	71.4		8.40	
SINGLE ANALYST REL DEV, %	3.04		7.63	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	5220.0	6468.0	24000.00	33600.00
MEAN RECOVERY (X)	4763.8	5716.3	24162.50	32787.50
% RECOVERY	91.26	88.38	100.68	97.58
OVERALL STD DEV (S)	562.0	740.1	1861.58	2296.21
OVERALL REL STD DEV, %	11.80	12.95	7.70	7.00
SINGLE ANALYST STD DEV, (SR)	330.3		1288.82	
SINGLE ANALYST REL DEV, %	6.30		4.53	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	1570.0	2200.0	160000.00	224000.00
MEAN RECOVERY (X)	1521.3	2062.5	136825.00	188875.00
% RECOVERY	96.89	93.75	85.52	84.32
OVERALL STD DEV (S)	209.2	159.9	17527.52	24567.27
OVERALL REL STD DEV, %	13.75	7.75	12.81	13.01
SINGLE ANALYST STD DEV, (SR)	159.6		17780.67	
SINGLE ANALYST REL DEV, %	8.91		10.92	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	14000.0	19600.0
MEAN RECOVERY (X)	12850.0	17725.0
% RECOVERY	91.79	90.43
OVERALL STD DEV (S)	838.4	937.7
OVERALL REL STD DEV, %	6.52	5.29
SINGLE ANALYST STD DEV, (SR)	643.4	
SINGLE ANALYST REL DEV, %	4.21	

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TABLE 53. STATISTICAL SUMMARY FOR ARSENIC,AS ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	8	8	8	8
TRUE CONC (C) UG/G	563.0	686.0	257.00	307.00
MEAN RECOVERY (X)	504.6	616.4	243.88	315.50
% RECOVERY	89.63	89.35	94.89	102.77
OVERALL STD DEV (S)	74.2	83.4	71.80	55.87
OVERALL REL STD DEV, %	14.70	13.53	29.44	17.71
SINGLE STD DEV, (SR)	45.1		46.93	
ANALYST REL DEV, %	8.05		16.73	
3-4 YOUDEN PAIR	**Solid 2A**	**2B**	**Solid 6A**	**6B**
NUMBER OF DATA POINTS	7	8	8	8
TRUE CONC (C) UG/G	293.0	363.0	37.00	45.00
MEAN RECOVERY (X)	262.6	351.5	28.66	37.81
% RECOVERY	89.61	96.83	77.47	84.03
OVERALL STD DEV (S)	59.5	86.0	9.86	13.60
OVERALL REL STD DEV, %	22.64	24.47	34.40	35.97
SINGLE STD DEV, (SR)	26.9		4.33	
ANALYST REL DEV, %	8.76		13.02	
5-6 YOUDEN PAIR	**Solid 3A**	**3B**	**Solid 7A**	**7B**
NUMBER OF DATA POINTS	7	7	8	8
TRUE CONC (C) UG/G	106.0	131.0	25.00	28.00
MEAN RECOVERY (X)	108.5	142.3	20.96	30.06
% RECOVERY	102.32	108.62	83.85	107.37
OVERALL STD DEV (S)	23.5	28.1	6.16	14.22
OVERALL REL STD DEV, %	21.70	19.76	29.37	47.32
SINGLE STD DEV, (SR)	9.7		6.11	
ANALYST REL DEV, %	7.74		23.96	
7-8 YOUDEN PAIR	**Solid 4A**	**4B**		
NUMBER OF DATA POINTS	8	8		
TRUE CONC (C) UG/G	233.0	237.0		
MEAN RECOVERY (X)	192.0	233.5		
% RECOVERY	82.40	98.52		
OVERALL STD DEV (S)	36.1	44.1		
OVERALL REL STD DEV, %	18.81	18.90		
SINGLE STD DEV, (SR)	18.3			
ANALYST REL DEV, %	8.59			

TABLE 54. STATISTICAL SUMMARY FOR SELENIUM,SE ANALYSES

1-2 YOUDEN PAIR	Solid 1A	1B	Solid 5A	5B
NUMBER OF DATA POINTS	7	7	8	8
TRUE CONC (C) UG/G	41.2	52.4	64.50	76.50
MEAN RECOVERY (X)	23.8	28.0	48.10	56.37
% RECOVERY	57.72	53.42	74.57	73.69
OVERALL STD DEV (S)	10.4	14.7	14.58	25.39
OVERALL REL STD DEV, %	43.77	52.67	30.32	45.04
SINGLE ANALYST STD DEV, (SR)	5.6		21.24	
SINGLE ANALYST REL DEV, %	21.45		40.65	

3-4 YOUDEN PAIR	Solid 2A	2B	Solid 6A	6B
NUMBER OF DATA POINTS	7	7	8	8
TRUE CONC (C) UG/G	22.0	27.0	102.00	127.50
MEAN RECOVERY (X)	13.5	17.9	68.27	97.42
% RECOVERY	61.40	66.12	66.94	76.41
OVERALL STD DEV (S)	6.3	9.4	19.53	25.33
OVERALL REL STD DEV, %	46.69	52.65	28.60	26.00
SINGLE ANALYST STD DEV, (SR)	3.1		14.46	
SINGLE ANALYST REL DEV, %	19.47		17.46	

5-6 YOUDEN PAIR	Solid 3A	3B	Solid 7A	7B
NUMBER OF DATA POINTS	6	6	8	8
TRUE CONC (C) UG/G	5.1	6.4	81.60	102.00
MEAN RECOVERY (X)	2.7	3.7	53.75	68.12
% RECOVERY	52.17	58.53	65.87	66.79
OVERALL STD DEV (S)	2.7	4.4	22.34	30.28
OVERALL REL STD DEV, %	99.47	117.01	41.57	44.44
SINGLE ANALYST STD DEV, (SR)	1.5		8.56	
SINGLE ANALYST REL DEV, %	45.30		14.05	

7-8 YOUDEN PAIR	Solid 4A	4B
NUMBER OF DATA POINTS	8	8
TRUE CONC (C) UG/G	32.5	40.2
MEAN RECOVERY (X)	18.0	20.9
% RECOVERY	55.36	51.99
OVERALL STD DEV (S)	12.6	13.0
OVERALL REL STD DEV, %	69.79	62.04
SINGLE ANALYST STD DEV, (SR)	2.3	
SINGLE ANALYST REL DEV, %	14.20	